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INTERIM PROGRESS REPORT

Carl V. Kanter, et al

Joint District, Federal and State Project for the Evaluation of Refinery Emissions

1 July 1956

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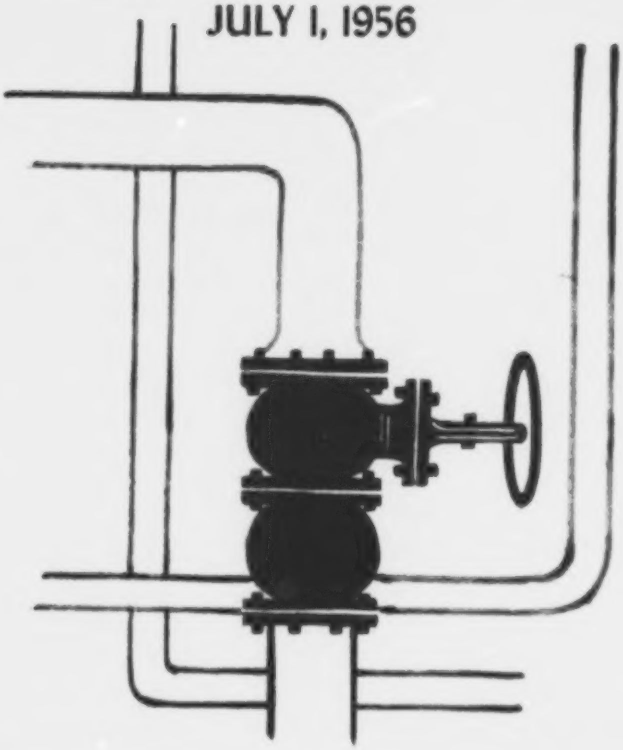

JOINT DISTRICT, FEDERAL AND STATE PROJECT FOR THE EVALUATION OF REFINERY EMISSIONS

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I

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FOREWORD

Primarily this report is addressed to the several agencies participating in the Joint District, Federal, and State Project for the Evaluation of Refinery Emissions. Its intent is to present the sponsors of the Joint Project with a detailed accounting of the progress made towards the evaluation of refinery emissions, recommendations, and a design for future work.

This report may serve to acquaint other agencies or groups interested in a similar program with useful sampling and testing techniques and survey methods. The results obtained by this Joint Project may indicate those sources of emission which warrant consideration in an air pollution control program.

SUMMARY

The Joint Project was initiated by the Air Pollution Control District of the County of Los Angeles to evaluate the emissions from the oil refineries in Los Angeles County. The California State Department of Public Health, the U.S. Public Health Service, and the Western Oil and Gas Association are participating in the Joint Project and providing, with the Air Pollution Control District, personnel for the Joint Project Committee and its testing and development program.

Out of several sources of possible emissions in refineries, six were selected for evaluation in the opening phases of the Joint Project. These were:

Catalytic cracking unit plumes
Cooling towers
Pumps
Valves and flanges
Storage tanks
Air blowing

Surveys were conducted to obtain information necessary for the planning of tests, extrapolation of test results, or as in the case of storage tanks, for the calculation of breathing, filling, and evaporation losses.

A Subcommittee on Procedures was given the responsibility for developing new sampling and analytical procedures or adapting existing procedures to the specific needs of the Project. The Subcommittee was also assigned direction of testing operations.

Despite the extensiveness of the program and the limited number of full time personnel, two items were completed and testing procedures for several others were developed and field tested. The two completed items were the calculation of breathing, filling, and evaporation losses from refinery crude and distillate storage tanks and the testing of Fluid Catalytic Unit plumes. Tentative procedures are outlined for the testing of cooling towers, pumps, valves and flanges, air blowing operations, and catalytic cracking regenerator stacks. A number of tests using these procedures have been performed.

The results of the surveys and calculations to determine losses from storage tanks are shown below.

TABLE I

Losses from Storage Tanks

Product	Number of Tanks	Capacity (barrels)	Hydrocarbon Losses (tons per day)
Crude oil	135	7,400,000	15
Light petroleum distillate	704	21,000,000	27
TOTAL	839	28,400,000	42

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RECOMMENDATIONS

Reliable estimates of the emissions of air contaminants from petroleum refineries and their contribution to community air pollution conditions are of importance not only to Los Angeles County but to any area where petroleum refining operations are conducted.

It is recommended, therefore, that the work of the Joint Project be continued in order to complete the studies already started and to examine other refinery sources marked for investigation.

This work may be completed in a year if sufficient personnel of requisite technical qualifications are assigned on a full time basis. At the present time only three persons are assigned full time to the Project. Although some additional assistance has been recruited from time to time on an "as needed" basis, this has not been sufficient to carry out the program expeditiously.

It is recommended that, in addition to persons now working on the project, the following personnel be assigned on a full time basis:

1. A source testing team consisting of three persons, two of whom are engineers.
2. A field team of two persons for direct measurement of emissions and collection of samples.
3. An analytical chemist for laboratory development of analytical procedures and analysis of samples.

INTRODUCTION

Approximately eight per cent of the oil refining capacity of the United States is concentrated within the boundaries of Los Angeles County. The capacity for processing over 700,000 barrels per day of crude oil makes it the third largest refining center in the nation (see Table II, pg.8). As a natural result of this, air pollution studies in the Los Angeles area have been concerned with the atmospheric contamination contributed by the petroleum industry. Investigations of emissions from petroleum refineries were intensified after the results of research pointed out the importance of hydrocarbons to smog formation.

Thorough surveys of each oil refinery were made by the Air Pollution Control District[1, 2] to enumerate sources of emissions and the types and quantities of contaminants emitted. The petroleum refineries individually undertook similar studies. Cooperatively, through the Western Oil and Gas Association, they retained Stanford Research Institute[3] to conduct basic research on the smog problem. In 1954 the Air Pollution Foundation

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[1] Unpublished Air Pollution Control District Survey Reports.

[2] Kanter, C.V., and Deckert, I.S., "Control of Air Pollution from Oil Refineries in Los Angeles County," Air Repair 4, 197 (1955).

[3] "The Smog Problem in Los Angeles County," Stanford Research Institute, a series of four reports, 1948, 1949, 1950, and 1954.

commissioned the Southwest Research Institute[4] to make an audit of refinery losses. While these studies were in progress, the petroleum industry proceeded with the installation of controls on known sources of contaminant emissions to effect a substantial reduction in losses to the air.

In 1955, both the Federal Government[5] and the State of California[6] initiated specific programs directed toward studies of air pollution problems. This mutual interest of the Federal Government, the State of California, and the Los Angeles County Air Pollution Control District prompted the formation of the Joint Project. Representatives of the petroleum industry were invited to participate in this Joint Project and to serve on the Joint Project Committee. They accepted this invitation and named, through the Western Oil and Gas Association, three representatives and two alternates to the Committee. Later, when it became evident that testing procedures were of extreme importance, they assigned, upon request, another representative to work on this problem.

---

[4] Swearingen, J. S., and Levin, H., "Hydrocarbon Losses from the Petroleum Industry in Los Angeles County," Air Pollution Foundation Report No. 5, 1954.

[5] "An Act to Provide Research and Technical Assistance Relating to Air Pollution Control," Public Law 159 - 84th Congress, Chapter 360 - 1st Session, S. 928.

[6] "Clean Air for California," California State Department of Public Health, March 1955.

In previous studies, the test data accumulated were meager; losses from many potential sources were merely estimated, and other sources were never evaluated. Consequently, the Joint Project Committee decided that this current evaluation of refinery emissions should be based on a testing problem comprehensive enough to insure that the results would be statistically valid. In cases where it is impossible to take test data, the estimate of losses to the atmosphere are to be based on the best possible methods of calculation after thorough studies of the equipment or operation under consideration.

TABLE II

Petroleum Refinery Operations in Los Angeles County

	Refinery Name	Approximate Crude Throughputs (Barrels/day)
1.	Calstate Refining Company	*
2.	Century Oil Company	5, 000
3.	Douglas Oil Company of California	6, 000
4.	Edgington Oil Refineries, Inc.	7, 000
5.	Envoy Petroleum Company	3, 000
6.	Fletcher Oil Company	5, 000
7.	General Petroleum Corporation	125, 000
8.	Hancock Oil Company	15, 000
9.	MacMillan Petroleum Corporation	8, 000
10.	Manespo Refining Company	4, 000 (estimated)
11.	Newhall Refining Company	3, 000
12.	Richfield Oil Corporation	130, 000
13.	Rothschild Oil Company	10, 000
14.	Shell Oil Company	80, 000
15.	Standard Oil Company of California	130, 000
16.	Sunset Oil Company	9, 000
17.	The Texas Company	60, 000
18.	Union Oil Company of California	125, 000
19.	Wilshire Oil Company, Inc.	25, 000
	Total	750, 000

*Cracking plant only; no crude oil processed.

ORGANIZATION

INITIATION OF THE JOINT PROJECT

Negotiations between the Air Pollution Control District and the Federal and State Governments for the initiation of a cooperative air pollution program were conducted at a series of three meetings--July 25, 26, and 27, 1955.

The outcome of these meetings was a decision to establish a joint study of petroleum refinery effluents in Los Angeles County. The Los Angeles County Air Pollution Control District was to direct the study and provide office space, transportation, test equipment, supplies, and most of the personnel. The State and Federal Governments were each to provide one man (both, however, to be assigned through the Department of Public Health, State of California) for a six month period beginning September 1, 1955. This initial assignment was later extended.

By mutual agreement, data developed during this evaluation could be used for publication by any of the three agencies, subject to the approval of the other two agencies. On August 4, 1955, a letter (see Appendix, pg.ii) was sent by Mr. Griswold, Air Pollution Control Officer of the Air Pollution Control District, to the Board of Supervisors of the County of Los Angeles recommending the establishment of a Joint Project. On August 9, 1955 an order was issued by the Air Pollution Control Board of Los Angeles County authorizing participation of the Air Pollution Control District in the Joint Project (see Appendix, pg.v).

Subsequently, a committee was appointed and a first meeting activating the Joint Project was held on September 16, 1955.

COMPOSITION OF THE JOINT PROJECT COMMITTEE

The joint Project Committee is composed of personnel from the Air Pollution Control District, the U.S. Public Health Service, and the Department of Public Health, State of California. Representatives of the Western Oil and Gas Association, by Mr. Griswold's invitation, also serve on the committee. This committee meets regularly to receive progress reports and outline future work.

Present membership of the committee is as follows:

Air Pollution Control District

Carl V. Kanter, Chairman	William E. Hart
Robert G. Lunche, Assistant to Chairman	Albert P. Fudurich
Raymond G. Holmes	Robert J. Bryan
George R. Cann	Stanley T. Cuffe*

State of California

Gerhardt C. Hass** - Bureau of Air Sanitation, Dept. of Public Health

Federal

Victor H. Sussman - Community Air Pollution Program, U.S. Public Health Service

---

*Mr. Cuffe was replaced by Mr. Robert C. Cofer on January 30, 1956.

**Mr. Hass was reassigned to duties at the Berkeley office and was replaced by Robert K. Palmer on June 1, 1956.

Petroleum Industry

Gerald Fisher	General Petroleum Corporation
Charles A. Jones	Shell Oil Company
Lawrence Richards	Richfield Oil Corporation
John H. Easthagen (alternate)	California Research Corporation (Richmond)
Joseph R. Scanlin (alternate)	The Texas Company
Davis A. Skinner*	Union Oil Company of California (Brea)

Of these, only Mr. Hass and Mr. Sussman have served full time on the Joint Project.

OBJECTIVE OF THE PROJECT

The over-all objective of the Joint Project is the determination of the quantity and nature of air contaminants being emitted as a consequence of petroleum refinery operations.

The program developed to attain this objective includes the following:

1. A study of all refinery operations with respect to equipment, products, operating conditions, and possible points of emission to the atmosphere.
2. Development or adaptation of sampling and analytical methods.
3. Field testing.
4. Evaluation, classification, and extrapolation of test results.

---

*Mr. Skinner was assigned in November, 1955, by the Western Oil and Gas Association to assist the committee in development of procedures.

PROGRAM

The following sources were selected by the Joint Project Committee for their program of investigation:

1. Cooling towers*
2. Air blowing operations*
3. Plumes from catalytic cracking units*
4. Valves, flanges, and other pipe connections*
5. Pipeline blind changing
6. Relief valves
7. Loading racks
8. Waste water separators
9. Storage tanks*
10. Pumps*
11. Catalytic cracking unit regenerators
12. Blowdown stacks
13. Waste gas flares
14. Vacuum jets
15. Shutdowns
16. Heater stacks

From previous studies it is known that the contaminants emitted to the atmosphere during petroleum refinery operations include hydrocarbons, oxides of nitrogen, particulate matter, sulfur compounds, ammonia compounds, and malodorous substances. Of these, the most important as far as smog formation is concerned are believed to be hydrocarbons and oxides of nitrogen. Major attention is being given to hydrocarbons.

---

*Selected for initial consideration.

SUBCOMMITTEE ON PROCEDURES

To develop the procedures necessary for carrying out the testing program, a Subcommittee on Procedures was appointed in November, 1955. The members of the subcommittee are:

Gerhardt C. Hass*
Victor H. Sussman
Davis A. Skinner
George R. Cann
Robert G. Lunche

The subcommittee was given the assignment of determining the sampling and analytical procedures to be used in testing the sources under consideration. As most of these sources had not previously been tested, standard procedures were not available.

This subcommittee work has been carried forward and several procedures have been prepared, some of which have been experimentally verified by laboratory and field tests. The sampling procedures are presented in the respective sections on Source Evaluations and the analytical procedures are in the Appendix.

In order to make use of the experience gained by the petroleum industry, their testing procedures were solicited by questionnaires. The responses pertaining to analytical methods for stack gases were tabulated by Mr. Fisher of General Petroleum Corporation and are shown in the Appendix, Table XIII. Those responses pertaining to testing procedures for other sources were tabulated by Mr. Skinner of Union Oil Company of California, and are shown in the Appendix, Table XIV.

---

*Mr. Hass was replaced on June 1, 1956, by Robert K. Palmer.

SOURCE EVALUATIONS

The following sections describe the progress made in the studies of each of the sources listed below:

Plumes from Catalytic Cracking Units
Cooling Towers
Valves and Flanges
Air Blowing
Storage Tanks*
Pumps*
Catalytic Cracking Unit Regenerator Stacks*

---

* No actual testing has been performed on these items.

PLUME TESTING

1. Sampling Procedure
2. Sampling Operations and Results

IN BRIEF:

Fluid catalytic cracking units on occasions are responsible for persistent plumes which form at a distance from the regenerator stacks.

Five sampling operations from a blimp* were performed in order to determine the nature of the particulate matter comprising these plumes. A high-volume Staplex sampler provided with fiberglass filters was used to capture the particulate matter. At the same time, samples were obtained for determination of oxides of nitrogen, carbon monoxide and hydrocarbons. Approximate dilution of the plume at the sampling location was to be determined from correlation of the carbon monoxide concentration in the plume and in the stack.

On only one of the five flights was a catalytic cracking unit plume present for sampling. The results were inconclusive.

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*The blimp was made available through the cooperation of the U. S. Naval Air Reserve Training Unit at Santa Ana, California

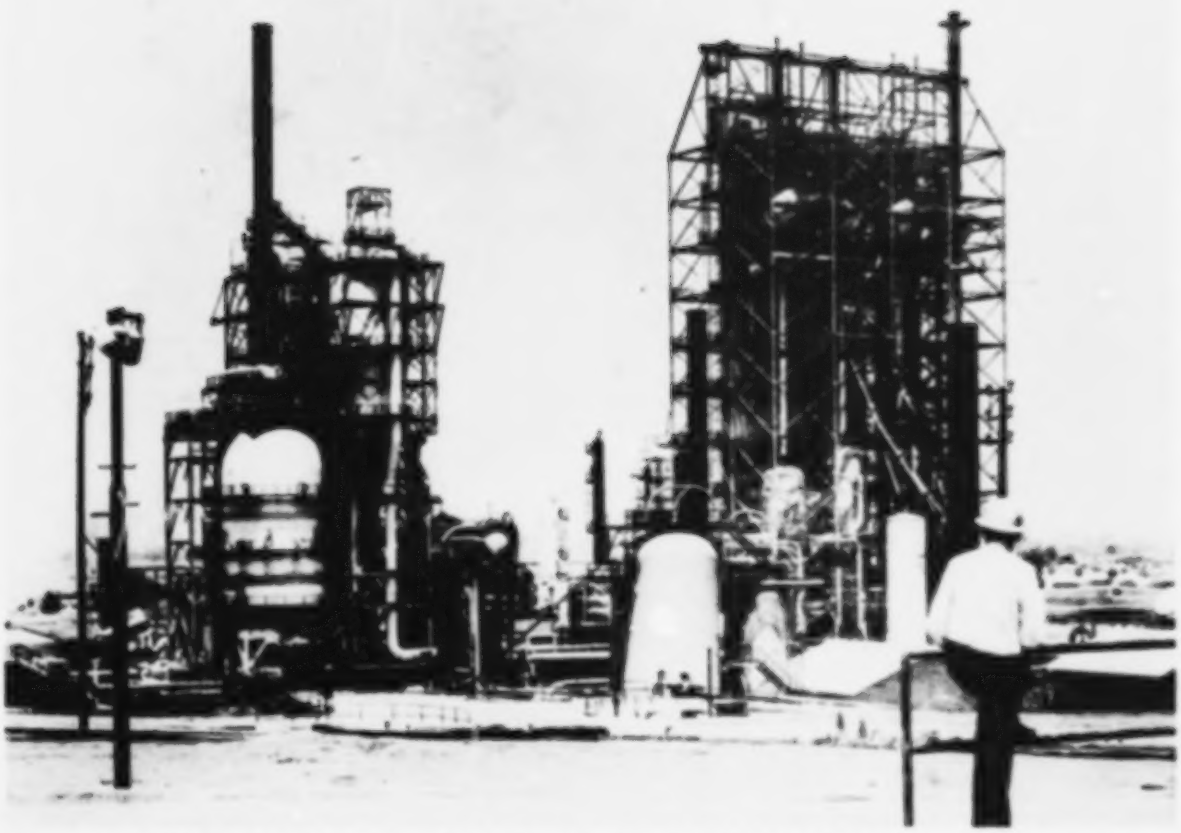

Figure 1...Catalytic Cracking Units. Fluid Catalytic Cracking Unit on left; Thermofor Catalytic Cracking Unit on right.

SAMPLING PROCEDURE
for
FLUID CATALYTIC CRACKING UNIT PLUMES
(Sampling from a blimp)

1. Scope

This method describes the procedure, using a blimp, for collecting samples for the determination of particulate matter, oxides of nitrogen, carbon monoxide and hydrocarbons in the plumes from catalytic cracking units.

2. Outline of Method

Samples of particulate matter are collected on a preweighed filter suspended beneath the blimp. The particulate matter is weighed and analyzed for ammonia and sulfur compounds.

Grab samples for the determination of oxides of nitrogen are collected in evacuated flasks containing Saltzman Reagent.

Carbon monoxide is measured directly by aspirating an atmospheric sample through an MSA Colorimetric Carbon Monoxide Tester.

Samples for hydrocarbon determinations are collected in five-gallon glass bottles by water displacement.

3. Apparatus and Materials

a. A Staplex high volume sampler, approximately 70 CFM capacity.

b. A gasoline motor-driven generator, 1 kilowatt, 110 volts, for furnishing power to the Staplex sampler.

c. Preweighed Mine Safety Appliance No. 1106, flash fired, fiberglass web filters and dust tight container.

d. Several 500 ml. evacuated bottles, wrapped with an opaque tape to keep out light and for safety, with self-sealing stoppers, and a hypodermic needle for puncturing stopper to permit inflow of sample. Each bottle contains 10 ml. of fresh Saltzman Reagent.

e. MSA Colorimetric Carbon Monoxide Tester, or equivalent, with supply of colorimetric tubes.

f. Several five-gallon bottles, several solid stoppers, one two-hole stopper, tubing for siphoning, six gallons of distilled water and a funnel.

g. Wet and dry bulb thermometers.

h. A reel with 400 feet of insulated electrical cord and electrical connections.

4. Sampling

Sampling is initiated when the blimp has been guided into proper position in or above the plume. Ground observers use two-way radio communication to guide the blimp into position. Sampling may be intermittent, depending upon the length of time that the blimp can keep the sampling equipment in the plume.

The Staplex Sampler is lowered by winch from the blimp's gondola. An electrical cable connects the Staplex Sampler to the power unit in the gondola. The sampler is equipped with a stabilizer fin and is weighted with sandbags to maintain a steady position, facing forward, during flight.

When the Staplex Sampler has been properly located in the plume during each pass of the blimp, the power is turned on. As the sampler exits from the plume after each pass, the power is turned off.

During the passes through the plume, wet and dry bulb temperatures and carbon monoxide readings are made and grab samples are collected for determination of oxides of nitrogen and hydrocarbons. For oxides of nitrogen samples a hypodermic needle is used to puncture the self-sealing stopper in the mouth of the 500-ml. evacuated bottle. A piece of tygon tubing attached to the hypodermic needle is used to draw a sample from a point forward of the motors. A period of about 30 seconds is required to fill the evacuated bottle with the air sample.

Samples for hydrocarbon determination are collected by siphoning water from a five-gallon bottle into an empty five-gallon bottle or drain. The sample is drawn in through tygon tubing from a point outside the blimp gondola forward of the motors. When all of the water has been siphoned out, the two-hole rubber stopper and tubing in the bottle are replaced by a solid stopper which is then wired in place.

Carbon monoxide is determined by aspirating an air sample through the MSA Carbon Monoxide Tester from a point forward of the motors outside the blimp gondola. Direct readings of the carbon monoxide content can be obtained by comparison of the color developed in the cartridge with the calibrated color scale on the Carbon Monoxide Tester.

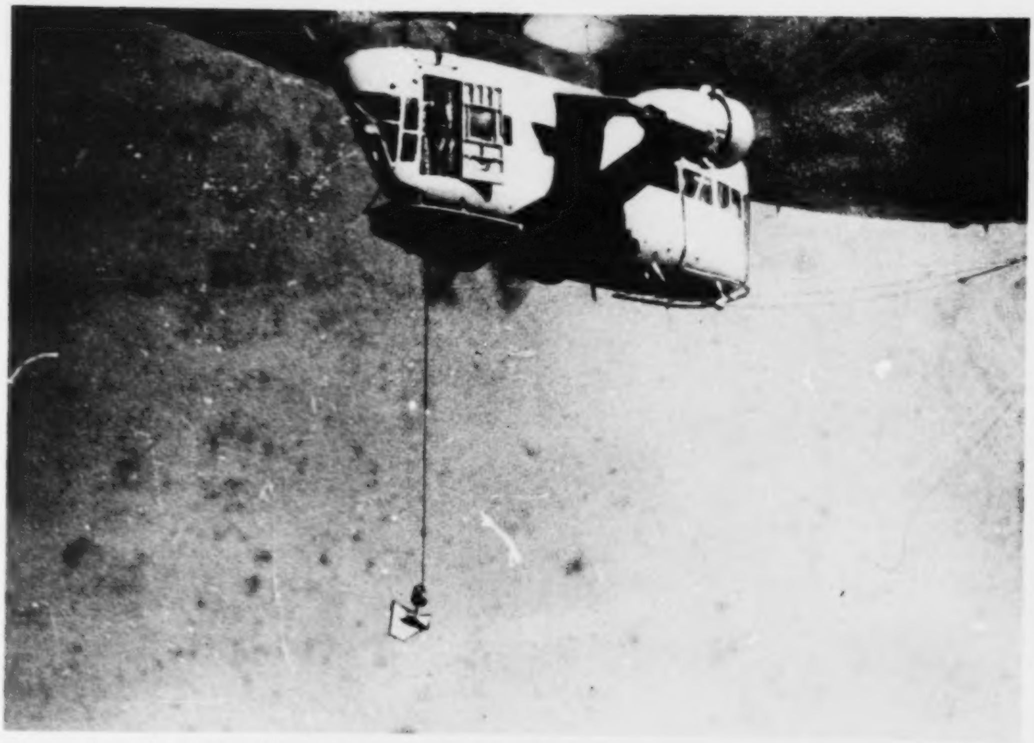

Figure 2...Blimp Gondola and Suspended Sampler

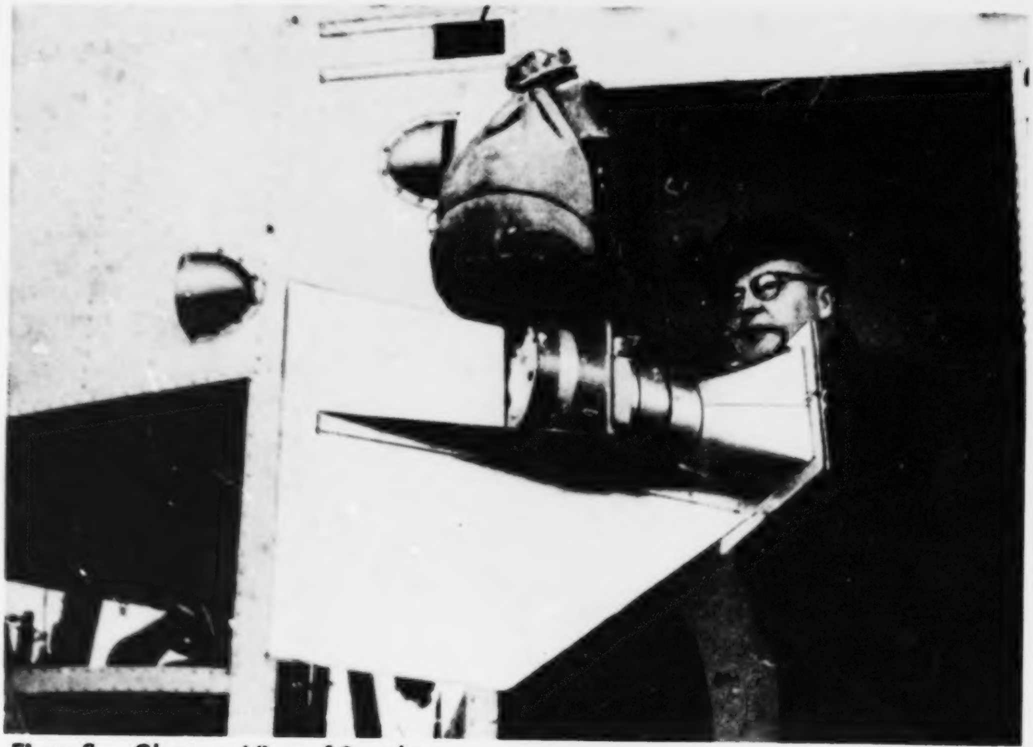

Figure3...Close-up View of Sampler

SAMPLING OPERATIONS AND RESULTS

During certain conditions of temperature and humidity the emissions from the regeneration of catalyst in a fluid catalytic cracking unit are attended by a visible plume. This plume forms at some distance from the top of the stack. It was felt desirable to gain some knowledge of the composition of the particulate matter in the plume in order to help postulate the mechanism of its formation.

The big problem in sampling this type of plume is its inaccessibility. The use of blimps, helicopters or captive balloons was suggested as a means of overcoming this problem. Blimps were selected because of:

1. their availability through the cooperation of the U. S. Navy*
2. their space for carrying test equipment and personnel.

A number of serious problems were encountered in the blimp flight program. The weather conditions most favorable to plume formation are generally unfavorable for blimp flights. Flight schedules could only be arranged for two or three days of the week, and the blimp could not be launched until 9:00 a.m., or until the weather had cleared. Thus, it was not always possible to make flights on days when conditions were favorable to plume formation.

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* U.S. Naval Air Reserve Training Unit, Santa Ana, California

The blimp had to maintain a speed of about 50 knots, could not approach too closely to the refinery structures, or to the ground, or safely trail the sampling apparatus more than about 50 feet below the gondola. It was thus difficult to obtain a sufficient size of sample for analysis, even after a number of passes through the plume.

Preliminary calculations were performed, using the Bosanquet-Pearson formula, to determine the dilution likely to occur in the plume. Employing values obtained from previous stack tests, it was concluded that a high-volume sampler should be used for capturing particulate matter.

Five blimp flights were made. Table III shows the dates of these flights and the sampling performed.

Table III

Record of Blimp Flights

Flight No.	Date	Type of Plume	Samples Collected for Determination of:
1	Nov. 4, 1955	FCC	Particulate matter, NO_2, CO, Hydrocarbons
2	Dec. 8, 1955	FCC	NO_2, CO, Hydrocarbons
3	Jan. 12, 1956	Power Plant	Particulate matter, NO_2, CO, Hydrocarbons
4	Jan. 20, 1956	FCC	Particulate matter, NO_2, CO
5	Feb. 10, 1956	Power Plant	Particulate matter

On only one sampling flight was a catalytic cracking unit plume present. On the other flights, when no catalytic cracking unit plumes were observed, general atmospheric samples were taken at varying altitudes upwind and downwind from the refineries. On the third and fifth flights power plant plumes were sampled.

Flight 1

No plume was observed during Flight No. 1, but sampling was attempted in order to test the equipment and technique.

Particulate Matter

The blimp circled the refinery at an elevation of 1000 feet (which was in the inversion layer) and approximately 3/4 mile from the Fluid Catalytic Cracking Unit. The Staplex Sampler was suspended 200 feet below the blimp gondola.

The sampler was run for 16 minutes at 65 CFM for a total volume sampled of 1040 cubic feet. The sampler ran continuously during the circling and therefore was possibly in the flue gas stream only a fraction of the sampling time.

The material collected on the filter was analyzed and the following was reported:

Total particulate matter collected - 0.010 grams
Total sulfur reported as SO_4 - 0.004 grams
No ammonia was detected.

Carbon Monoxide

Tests for concentrations of carbon monoxide were made with a MSA Colorimetric CO tester. The inlet port of the tester was placed outside of the blimp gondola, forward of the engines. This tester measures CO concentrations in the range of 0.005 to 0.10% by volume. Greater sensitivity may be obtained by making two or five aspirations instead of one. Since the blimp was moving through different ranges of concentration the bulb was aspirated only once for each detector tube.

A palpable odor was noted at a location 3/4 of a mile S. E. of the FCC unit. A concentration of 100 ppm was recorded on the CO tester while in the region of the odor.

The intensity of the odor and relatively high concentration of carbon monoxide at this distance from the FCC unit may be explained by the fact that the blimp was sampling in the inversion layer. The strong thermal updraft from the FCC unit very likely carried the stack gases into the inversion layer, where the stable atmosphere retarded their diffusion.

Oxides of Nitrogen

The concentrations of NO_2 in the samples taken S. E. of the FCC unit were found to be slightly higher than those in the samples taken at other points.

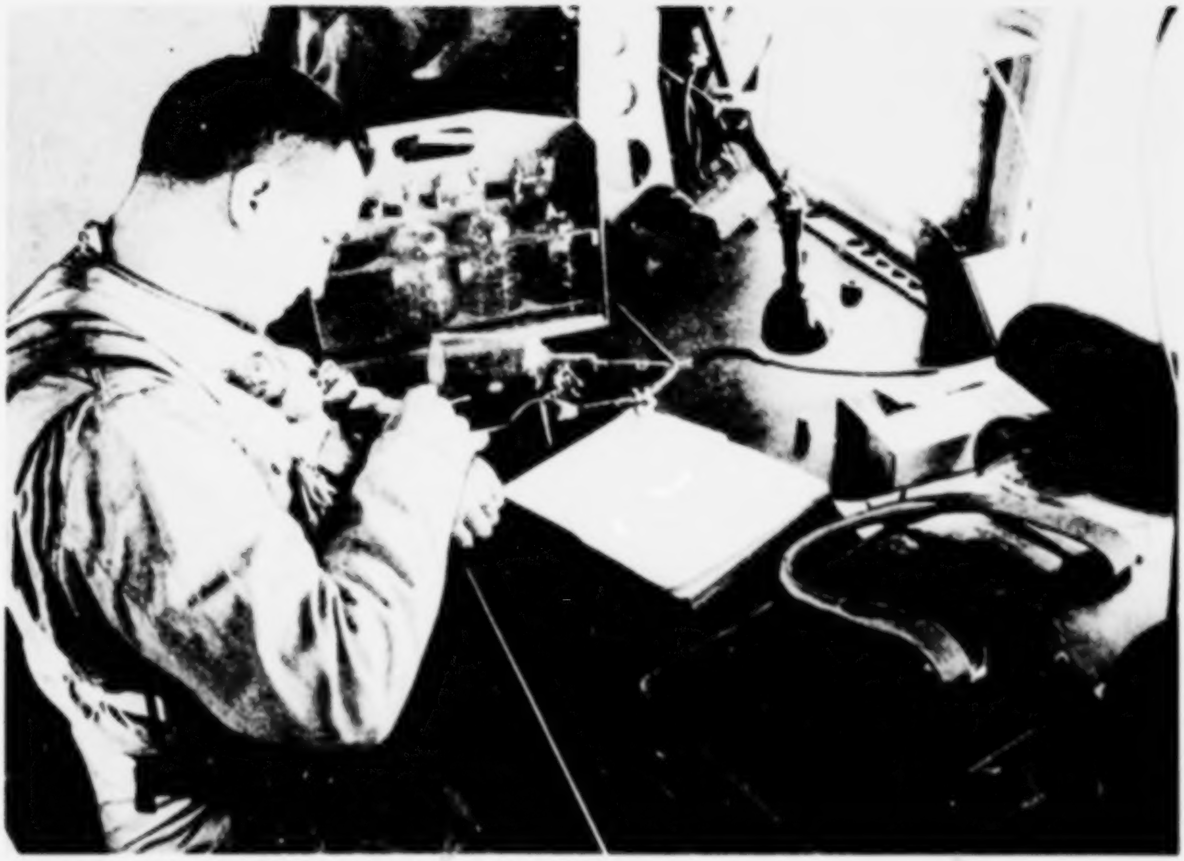

Figure 4...Obtaining Oxides of Nitrogen Grab Samples

Table IV

Concentration of NO_2, Blimp Flight 1

Concentration of NO_2 (ppm)	Position of Blimp Relative to FCC Unit
0. 39	SSE
0. 36	N
0. 25	NW
0. 39	SE

Hydrocarbons

Samples for hydrocarbon analysis were taken in 500 ml. evacuated bottles. It was found that the hydrocarbon concentration was below the sensitivity of the I. R. analyzer for the quantity of sample collected.

Flights 2 and 3

During Flights 2 and 3, no plume was visible when the blimp arrived at the sampling location. General atmospheric sampling was conducted at various altitudes upwind and downwind of the refineries. The results are shown in Tables V and VI. On Flight 2, there was no sampling for particulate matter.

Particulate Matter

On Flight 3, an attempt was made to sample the plume at a power plant. The Staplex Sampler was directed into the power plant plume and the blimp traversed the plume starting at a downwind point. Analysis of the filter paper from the Staplex Sampler yielded no results. It was doubtful that the Staplex Sampler was in the plume during this run since ground observers were not in a position to view from the side.

Hydrocarbons

Samples for hydrocarbons were collected in five-gallon bottles by water displacement, to provide larger samples than were taken in Flight 1.

Tables V and VI show that the hydrocarbon concentrations, determined during Flights 2 and 3, were generally higher downwind of the refinery than upwind.

Table V
Results of Air Sampling, Blimp Flight 2, December 8, 1955

Time	Location	Elevation (Above Sea Level) Feet	Wind at Ground Level Speed MPH	Wind at Ground Level Direction	Temperature Wet Bulb °F.	Temperature Dry Bulb °F.	Rel. Hum. %	Analytical Results NO$_2$ ppm	Analytical Results Hydrocarbons ppm	Analytical Results Carbon Monoxide %
10:55 AM	1/2 mile So. of Refinery C.	925	0-4	N				0.02	0.39	<0.005
11:04-11:05		1700	3-4	N				0 0.04	0	<0.005
11:14-11:22		500	3-4	N	50	68.5	23	0.06 0.02 0.01 0.07	0.35	<0.005
11:20-11:22		Ground	<3	N	53	74	21	0.13	0.13	<0.005
11:26-11:28	1/4 mile No. of Refinery C	900	3	N	48	66	23	0.05 0.03	0.22	0.005
11:34-11:36		500	3	N	50	68.5	23	0.04 0.06	0.23	<0.005
11:47-11:48		1500	3	N	45	65	13	0 0.08		<0.005
11:55-11:58		1900						0.05 0.01		<0.005
11:40-11:44		Ground	<3	N	57	75	31	0.04	0.09	<0.005
12:08-12:11	1/4 mile So. of Refinery B	900	3	N	49	68	21	0.09 0.06		<0.005
12:40 PM		Ground	0		59	81	25	0.02	0	<0.005
12:19	1/4 mile No. of Refinery B	960	3	N	48.5	68	19	0.06		<0.005
12:20-12:24		Ground	<3	N	55	74	30	0.07	0.09	<0.005

Table VI

Results of Air Sampling, Blimp Flight 3, January 12, 1956

Time PM	Location	Elevation (Above Sea Level) Feet	Wind at Ground Level		Temperature		Rel. Hum. %	Analytical Results		
			Speed MPH	Direction	Wet Bulb °F.	Dry Bulb °F.		NO_2 ppm	Hydrocarbons ppm	Carbon Monoxide %
12:40-12:48	1/2 mile SE of Refinery B	2100	5	NW	55	66	50	0.05 0.19	1.60	<0.005
12:59- 1:08		625	3	NW	59.5	70	55	0.33 0.16	1.26	"
1:12- 1:22		425	2-3	NW	60	72	50	0.18 0.19	0.35	"
12:44-12:53	SSE of Refinery B	Ground	1-2	SE	62	73	55	0.33	0.22	"
1:52- 2:01	1/4 mile NNE of Refinery B	1400	10	NW	57 53	65 68	62 46	0.06 0.09	0.52	"
1:40- 1:48		700	3	NW	59	70	53	0.15 0.07	0.30	"
1:28- 1:37		400	3	NW	58	69	52	0.18 0.17	0.83	"
1:28	NE of Refinery B	Ground	0-1	SW (var.)	60.5	72.5	51	0.15	0.70	"
1:47- 1:52		Ground	0-1	SW (to W)				0.09	0.61	"
2:10- 2:15	E of Refinery B	Ground	0-3	SW	61	74	47	0.14 0.22	0.61	"

Table VII shows the corresponding upwind and downwind concentrations at selected altitudes.

No controls or blanks were run on this method in order to determine its precision or accuracy. The determination of hydrocarbon concentrations by this method was carried out only during Flights 2 and 3.

Table VII

Atmospheric Hydrocarbon Concentrations

	Elevation ft.	Upwind Concentration of Hydrocarbons ppm	Downwind Concentration of Hydrocarbons ppm
Flight 2 Refinery C	0	0.09	0.13
	500	0.23	0.35
	900	0.22	0.39
Flight 2 Refinery B	0	0.09	0.0
Flight 3 Refinery B	0	0.22	0.7
	400	0.83	0.35
	600-700	0.30	1.26
	1400-2100	0.52	1.60

Flight 4

The only time the Staplex Sampler was able to operate within a dense F. C. C. plume was during Flight 4. This flight was made on a drizzly day. During the sampling run, the blimp was at an altitude of about 350 feet above the ground and the Staplex Sampler was suspended about 50 feet below the blimp gondola. The blimp made several passes through the plume with a total sampling time of 7 minutes 36 seconds. The sampler operated at 65 CFM.

In making these runs, the blimp went downwind (the wind was from the south) about one half mile from the refinery and to the west of the plume. It then circled and flew directly along the path of the plume upwind until it approached within approximately 100 yards of the catalytic cracking unit. At that point it veered off to the east, circled the refinery and repeated its flight along the path of the plume.

Observers on the ground to the west and east of the plume and one directly underneath the plume assisted in orienting the blimp by radio communication. During the sampling runs it appeared to both personnel in the blimp and ground spotters that the sampler was actually in the plume. Visibility was poor due to the overcast.

The preweighed filter was tested for increase in weight and for acidity. Both tests yielded negative results.

Assuming that particulate matter is a constituent of the visible plume, these negative results could be due to the plume being diluted to a greater extent than anticipated. The wetting of the filter from the drizzle may have washed off any material collected or dissolved it and washed it through the filter.

The filter paper has an exposed area of 7 in. x 9 in. Operating at a rate of 65 CFM the face velocity is 149 linear feet per minute. During the sampling, the sampler traversed the plume upwind. The average wind speed during the tests was approximately 3 knots and the blimp was traveling at an average speed of 40 knots. It is possible to introduce large errors in sampling for particulate matter by deviating from isokinetic conditions. From an examination of the above, it is obvious that at no time during the sampling were isokinetic conditions achieved.

Flight 5

There was no visible FCC plume on this flight and so a power plant plume was sampled for particulate matter. This plume rose to about 1200 feet and then drifted horizontally to the west. Ten passes through the plume were made with the Staplex Sampler operating 60 feet below the gondola for a total sampling time of 22 minutes.

The exposed portion of the filter paper showed a tan discoloration but a weighable amount of material was not obtained.

COOLING TOWERS

1. Sampling Procedure
2. Sampling Operations and Results
3. Revision of Sampling Procedure

IN BRIEF:

Large quantities of water are used in petroleum refineries for cooling. The heat absorbed by the water must be removed before the water can be reused. This is accomplished by evaporation and convection as the water cascades over baffles in cooling towers. Hydrocarbons may enter the water from leaks in process equipment, be carried to the cooling tower and there lost to the atmosphere.

In order to determine emissions from this source, surveys were conducted to obtain the number, water rates and types of service of cooling towers. Nine cooling towers were tested. The results obtained to date indicate that hydrocarbon losses do occur from this source, but the total amount lost has not yet been determined.

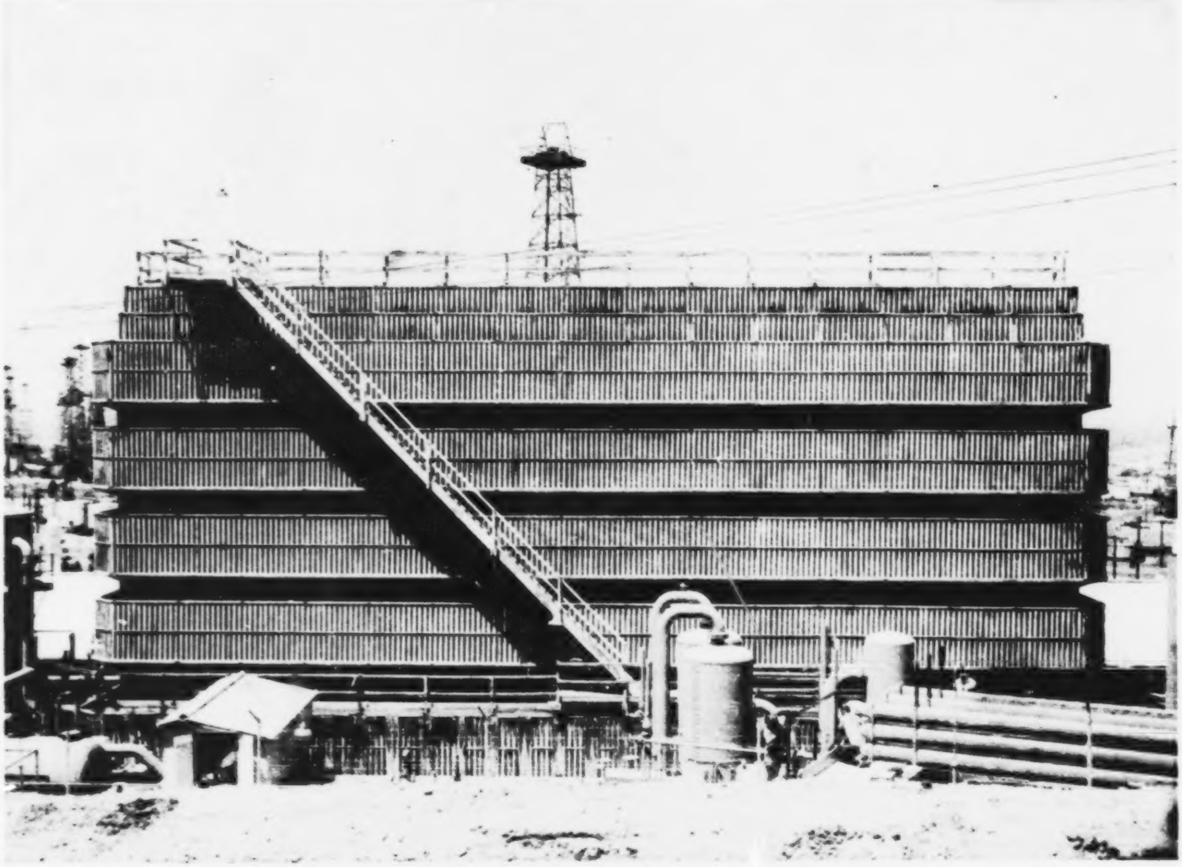

Figure 5...Cooling Tower

SAMPLING PROCEDURE
for
COOLING TOWERS

1. Scope

This method describes a procedure for the determination of hydrocarbon losses to the atmosphere from refinery cooling towers.

This method is not applicable to cooling towers having exchanger bundles within the tower proper or open condenser boxes on the lines leading to the tower.

2. Outline of Method*

A continuous, metered sample of spent cooling water is passed through a settling vessel, where liquid and gaseous hydrocarbons are allowed to separate from the water phase. At the end of the sampling period the volume of the gas phase is measured and the concentration of hydrocarbon is determined by infrared spectrophotometry or by a combustible gas indicator. The liquid hydrocarbon phase is removed from the settling vessel and the low-boiling fraction is removed by distillation. The weight of this fraction is added to the weight of the gaseous hydrocarbons.

3. Apparatus and Materials

a. Water meter, integrating type

b. 13-gal. carboy

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* This method is being revised. See page 48

c. Copper tubing, 1/4 in. and 1/2 in.

d. Rubber rubing, hose clamps, screw clamps, and 1/4 in. I. D. rubber pressure hose.

e. Thermometer, to 212° F.

f. Mercury manometer

g. Graduated cylinder, 2-liter

h. Pipette, 100 ml.

i. Combustible Gas Indicator

j. Drierite tube

k. Two 2-liter evacuated bulbs

l. 250-ml. sampling bottle

4. Sampling

The sampling tap is located at a point in the cooling water return line where stratification is at a minimum, e.g., a vertical riser or a tap on a centrifugal pump casing. The apparatus is connected as shown in Fig. 6. The carboy is filled with water until a vapor space of about two gallons remains. The inlet tube is bent so that the water entering impinges on the wall of the vessel above the liquid level. The clamp on the gas take-off hose is closed. The sampling valve is open full. If the flow rate is greater than one gallon per minute, the rubber tubing on the inlet should be constricted by tightening the screw clamp on the pressure tubing. After a flow rate of one-half to one gpm is obtained, the outlet tubing is adjusted to a height such that a slight positive pressure is maintained in the vapor space, as determined with the mercury manometer attached to the gas outlet tube. Water meter readings are taken at the beginning and end of the sampling period.

The sampling is carried out for a period of 7 days. The liquid level in the carboy is observed daily during the sampling period. If the liquid level is reduced below the halfway mark on the carboy, the test is interrupted and the gas phase is sampled as described below. The remaining gas is expelled and the test is resumed to complete the 7-day period.

DIAGRAM OF COOLING TOWER SAMPLING EQUIPMENT

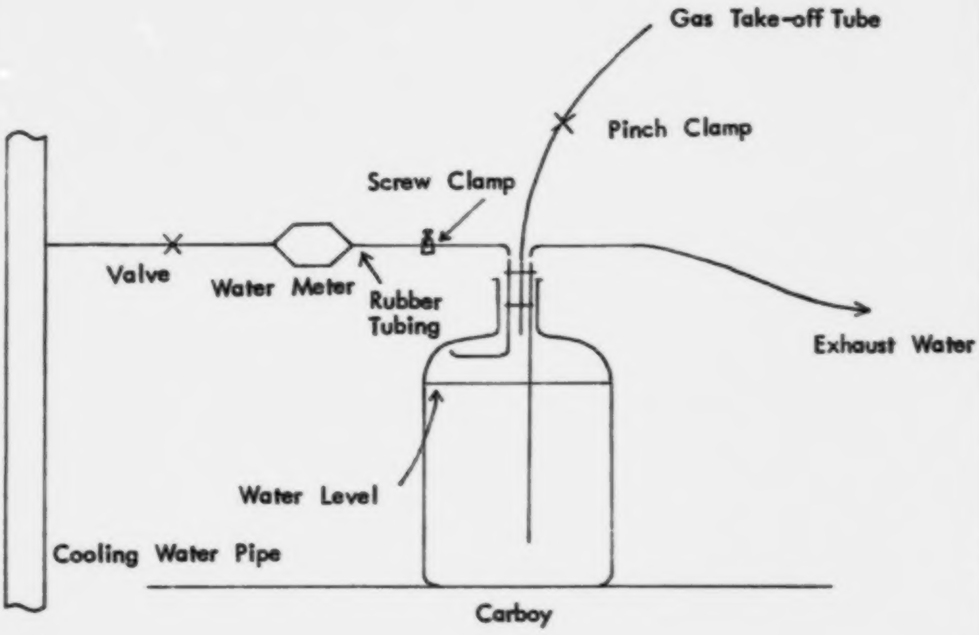
Gas Take-off Tube
Pinch Clamp
Screw Clamp
Valve
Water Meter
Rubber Tubing
Exhaust Water
Water Level
Cooling Water Pipe
Carboy

Figure 6

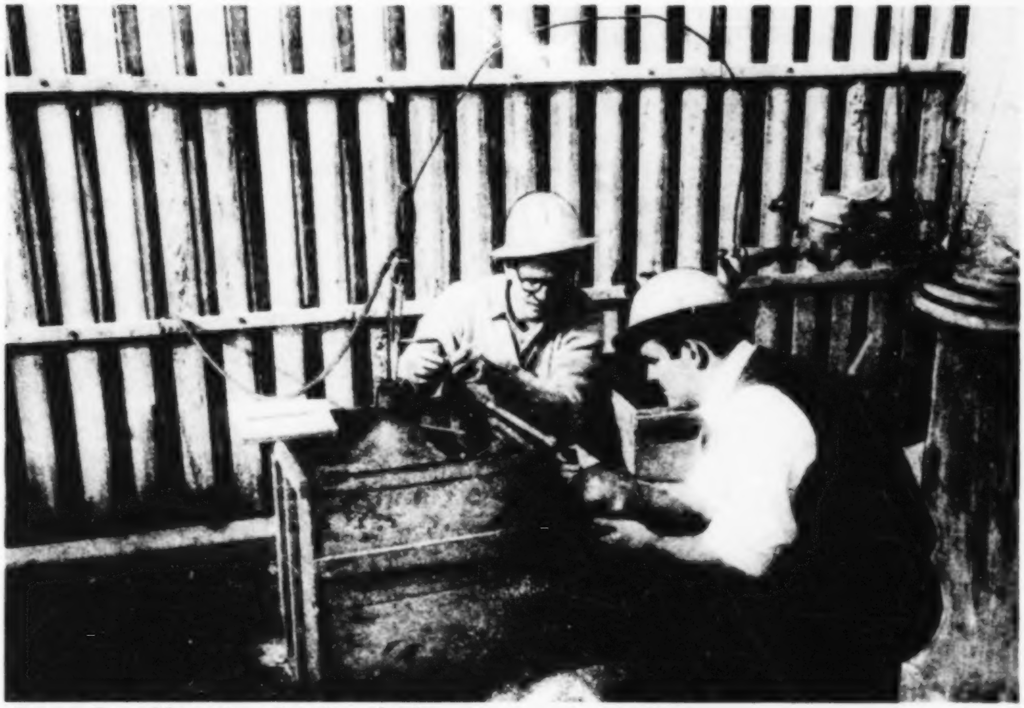

Figure 7...Obtaining Gas Sample from Cooling Tower Water

a. Gas phase sampling

At the conclusion of the sampling period the liquid level in the carboy is marked, the temperature of the effluent water is measured and the pressure in the gas space is noted. The gas volume is later determined in the laboratory by filling the carboy from the mark with a measured volume of water.

(1) The gas take-off hose is connected to the combustible gas indicator through a Drierite tube. The meter is purged and the concentration of hydrocarbons in terms of

percentage of lower explosive limit is recorded. If the meter reading is on-scale, this reading is used to determine the hydrocarbon concentration in the gas phase.

(2) If the reading is off-scale, it is necessary to take a sample for infrared analysis. A 2-liter evacuated sampling bulb is used.

b. Liquid phase sampling

If a hydrocarbon layer is observed in the carboy, water is added through the water outlet tube to float the hydrocarbon layer to the neck of the carboy. The hydrocarbon is then pipetted into the liquid sample bottle. The sample is later distilled and the weight of distillate collected up to a temperature of 160° F. is determined.

5. Calculations

The concentration of hydrocarbon in the gas phase is expressed as ppm by volume of n-hexane when determined by the infrared spectrophotometer. If the combustible gas indicator reading is used, the concentration of hydrocarbon is taken from the graph shown in Fig. 8 expressed as per cent by volume of n-hexane. These values are converted to express the hydrocarbon concentration in terms of weight per standard cubic foot.

The weight of hydrocarbon in the gas phase is calculated from the concentration and the gas volume, corrected for pressure and temperature.

This weight is added to the weight of the 160° F. distillate to give the total weight of hydrocarbons.

Sample calculation:

Cooling water rate	6,000 gpm
Sample water rate	1.03 gpm
Sample time	18-1/3 hrs.
Water temperature	101° F. (561° R)
Gas pressure	0.2 in. Hg gauge (30.1 in. Hg absolute)
Gas volume	1.47 cu. ft.
Hydrocarbon concentration	5.2% as n-hexane
No liquid hydrocarbon collected.	
Molecular weight of hexane	86

$$.052 \times 1.47 \times \frac{86}{359} \times \frac{492}{561} \times \frac{30.1}{29.9} = .0162 \text{ lbs. hydrocarbon in gas phase}$$

$$.0162 \text{ lbs.} \times \frac{6000 \text{ gpm}}{1.03 \text{ gpm}} \times \frac{24 \text{ hrs./day}}{18\text{-}1/3 \text{ hrs.}} = 123 \frac{\text{lbs.}}{\text{day}} \text{ hydrocarbon loss from tower}$$

FRACTION OF LOWER EXPLOSIVE UNIT
VS
PER CENT BY VOLUME IN AIR

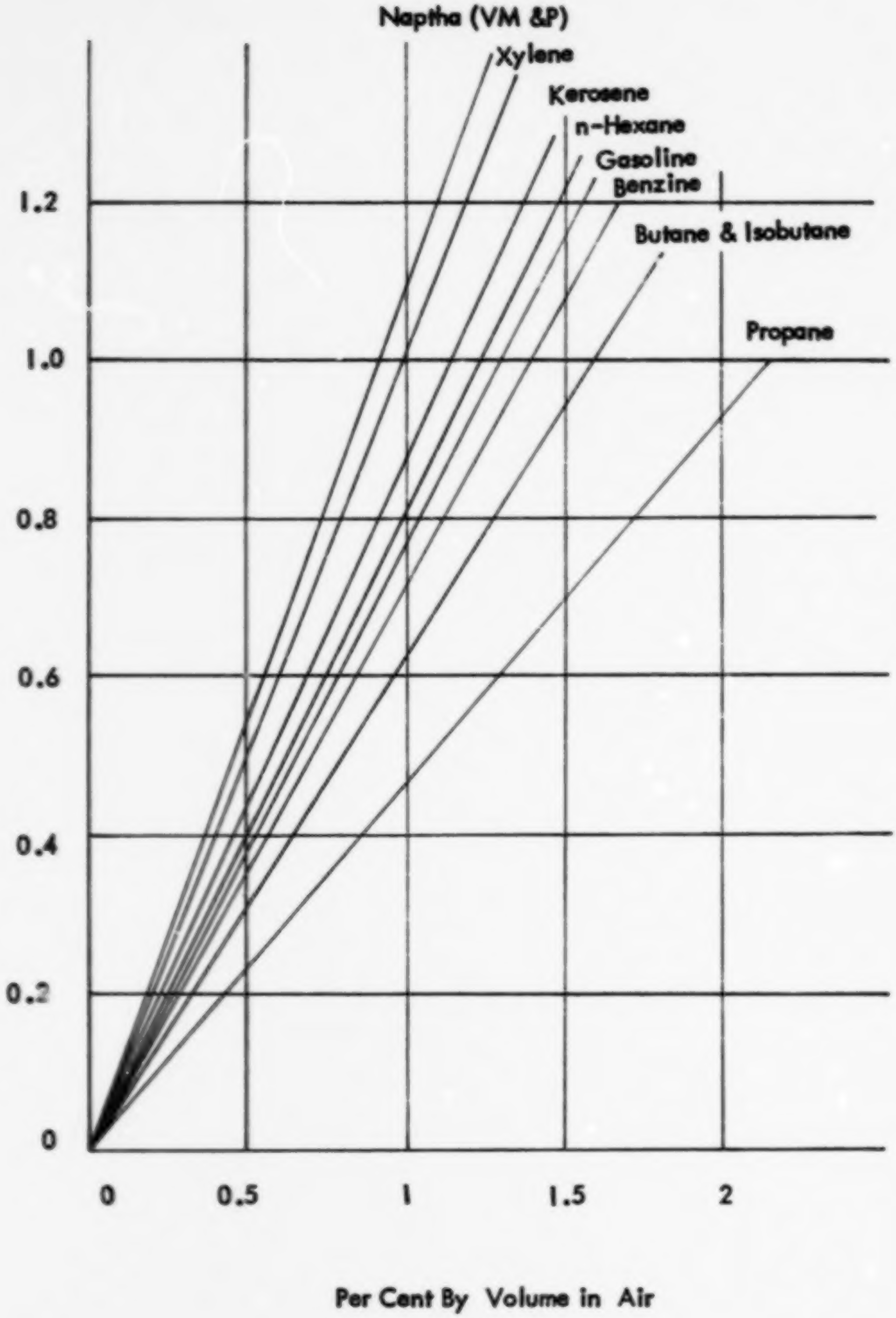

Figure 8

SAMPLING OPERATIONS AND RESULTS

The cooling tower testing program was designed to measure hydrocarbon losses to the atmosphere from process cooling waters. These waters are cooled for re-use by evaporation of a portion of the water when passing over large cascade structures. Hydrocarbons may enter the water through leaks in heat exchangers, condensers, etc., and subsequently escape to the atmosphere at the cooling tower.

In early discussions about cooling tower testing, there were suggestions to sample the atmosphere downwind of the cooling tower and its plume. It was felt that this was inadvisable for the following reasons:

1. The minute concentration of hydrocarbons likely to be present and the large amounts of water vapor in the plume would make sampling and analysis difficult.

2. It is impossible to select a representative sampling point. The plume is emitted from various levels of the forced draft and natural draft towers. The concentrations of the contaminants in this plume, at the various levels, are not likely to be constant. The only type of tower where a representative sampling point may be found is a single fan, induced draft tower. If this type of tower were to be sampled a velocity traverse would be required.

Information was obtained on the 86 cooling towers in service in the

Los Angeles County refineries for cooling process waters. From this data, cooling towers were selected to be tested. Nine tests were performed on six different cooling towers. The test data and calculated results for these tests are presented in Table VIII.

These data are subject to a serious limitation as a result of two errors inherent in the method of sample collection. The sampling procedure was based on the assumption that hydrocarbon loss from the carboy due to solubility in the exhaust water was negligible. Calculations have since shown that there may be an appreciable loss due to the solubility of hydrocarbon in water. Preliminary work has also shown that minute droplets of hydrocarbon may remain suspended in the body of the water for a period of 6 hours or more. Thus, in the method used, these droplets are carried out and lost.

The data shown in Table VIII under the column "Hydrocarbon Loss to Atmosphere" are derived from the amount of hydrocarbon collected in the gaseous phase in the carboy. In the test on tower A-13 about 70 grams of liquid hydrocarbon was also collected in the carboy. The liquid was removed from collected sludge by steam distillation. If all of the liquid represented by this amount were lost to the atmosphere during a single pass through the cooling tower, the additional hydrocarbon loss from tower A-13 would be 217 pounds per day.

Table VIII

Cooling Tower Test Results

Cooling Tower	Cooling Water rate gpm	Sample Water rate gpm	Sampling time hrs.	Water Temp. °F.	Gas pressure in. Hg	Gas volume cu. ft.	Explo-simeter Reading % of L.E.L.	I. R. Analysis % as n-hexane	Hydrocarbon loss to atmosphere lbs. /day
A 14-1	12,000	0.82	52-1/4	100	0.2	1.60	0.2		4.6
A 14-2	6,000	1.03	18-1/3	101	0.2	1.47	>1.2	5.2	123
B 6-1	3,000	0.69	92-1/2	88	0.4	0.80	>1.2	0.56	1.1*
B 6-2	3,000	0.68	171-3/4	80	1.3	1.07	0.45		<1
B 7-1	14,000	0.87	92-3/4	92	2.1	1.60	1.1	7.3	111
B 7-2	14,000	0.87	171-1/2	91	2.9	1.47	>1.2	8.9	69
CT	12,600	0.59	192-1/2	104	4.0	0.93		0.002	<1
CS	3,000	0.61	168	83	1.0	0.80	0.1		<1
A-13	6,300	0.77	139-1/2	90	0.2	1.53	>1.2	2.0	9.3**

---

* I. R. value used in calculation.

** Does not include 70 grams of liquid hydrocarbon collected in carboy.

REVISION OF SAMPLING PROCEDURE

The revised cooling tower sampling procedure will consist of taking periodic grab samples from both the inlet and outlet of the cooling tower for hydrocarbon determination. The samples will be stripped with nitrogen gas which is then passed through a freeze-out trap and the hydrocarbons thus collected will be analyzed by infrared spectrophotometry.

Fig. 9 shows the equipment used in the laboratory to strip the cooling tower water samples and collect the hydrocarbons. Shown from right to left are the compressed nitrogen supply tank, bubbler, rotameter, drierite tube and freeze-out trap in a flask of liquid oxygen. On the table are shown a spare bubbler, flask for liquid oxygen and freeze-out trap.

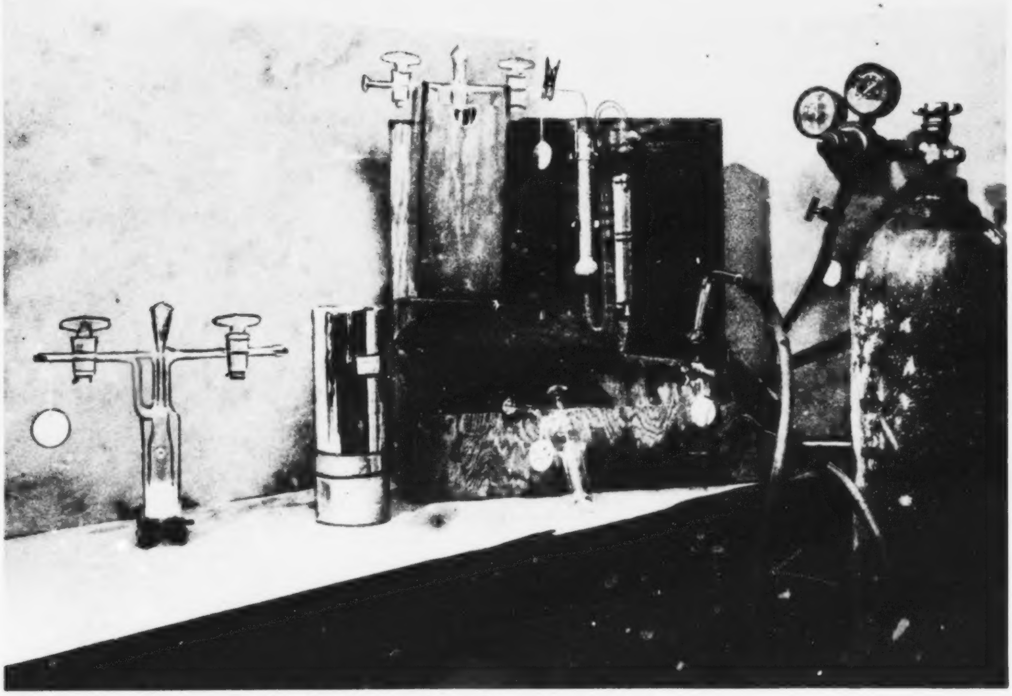

Figure 9...Equipment for Analysis of Cooling Tower Water

VALVES AND FLANGES

1. Sampling Procedure
2. Sampling Operations and Results

IN BRIEF:

Leaks from valves and flanges may be responsible for appreciable emission of hydrocarbons to the atmosphere.

Surveys were made to obtain the number of valves and flanges, classified by service, in the refineries. A testing program was initiated to obtain leakage rates from a representative number of these units.

Four sampling operations were carried out. Of 394 valves tested (representing every sixth valve of 2, 364 counted) 42 leaks totalling 244 lb/day of hydrocarbons were found.

Additional tests must be run in order to obtain sufficient data for statistically evaluating the total emissions to the atmosphere from this source.

Flanges appear to be of minor importance.

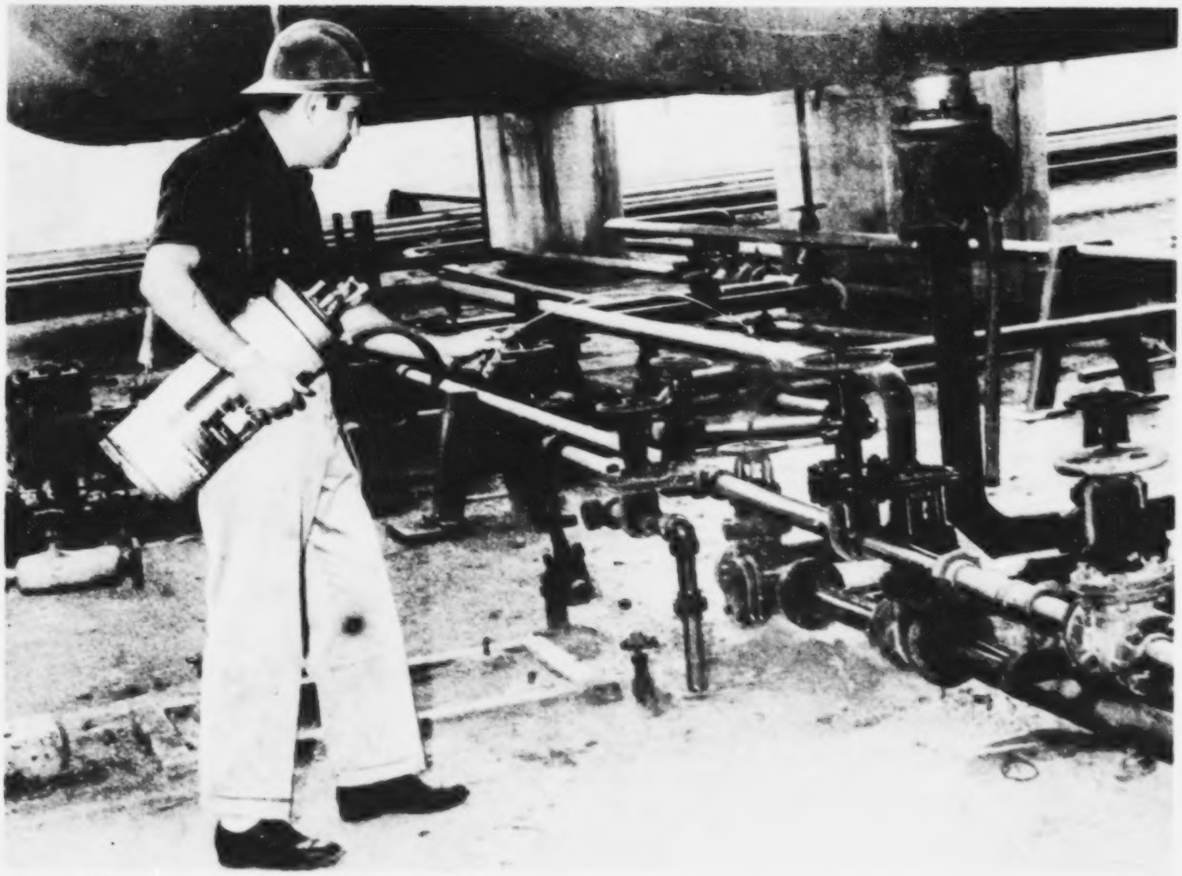

Figure 10...Spraying Valve to Check for Leakage

SAMPLING PROCEDURE
for
VALVES AND FLANGES

1. Scope

This method describes procedures for the measurement of hydrocarbon losses from leaking valves and flanges. Both liquid and gaseous leaks are included.

2. Outline of Method

A leaking valve or flange is encased in an airtight shroud. The rate of gas leakage is measured by a wet test meter. A liquid leak is measured directly by collection in a graduated cylinder.

3. Apparatus and Materials

a. Polyethylene sheeting - approx. 4 ft. wide and 3-5 mils in thickness.

b. Wet test meter

c. Polyethylene and rubber tubing - 1/4 inch I. D.

d. Glyptal cement

e. Masking tape - 1 and 3 inch widths.

4. Procedure

It is first necessary to locate those valves and flanges that are leaking. A liquid leak is found by observation; a gaseous leak by spraying the fittings with a soap solution and observing the production of bubbles.

The fitting is then encased in the polyethylene sheeting. Masking tape is used to bind the seams of the enclosure. Pleats or wrinkles along the

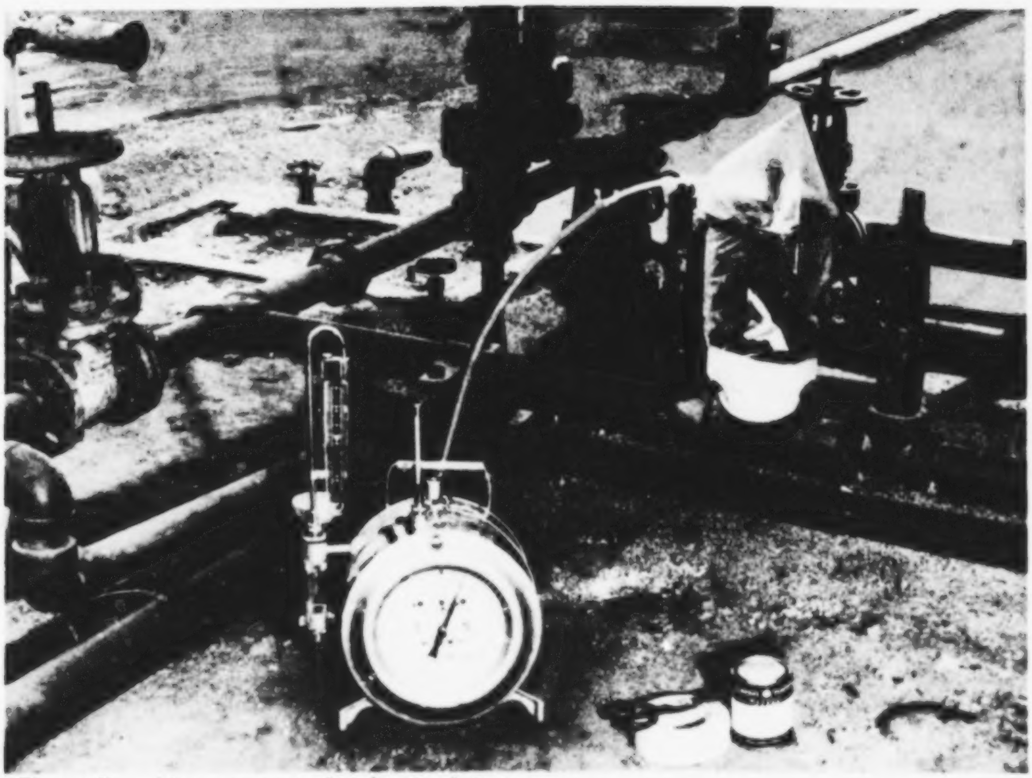

Figure II...Measurement of Valve Leak Rate

seam should be avoided. Glyptal cement is used to seal the edge of the polyethylene at the line of contact with the pipe. The glyptal is applied to the pipe and the edge of the sheeting placed over it. Masking tape is then placed over the edge of the polyethylene sheet and in contact with the pipe.

A short length of 1/4 in. I. D. polyethylene tubing is sealed into the shroud at one of the seams (preferably at a corner). A length of 1/4 in. I. D. rubber tubing is used to conduct the gas liberated at the leak from the polyethylene tubing to the meter.

It is advisable to test the shroud for leaks before attaching the rubber tube to the meter. This is accomplished by gently blowing back into the shroud through the rubber tube. The shroud is painted with soap solution so that leaks, if any, may be detected.

The system is allowed to reach a steady state and the rate of gas leakage is recorded. The temperature and pressure at the wet test meter are recorded.

Liquid leaks are measured by collection in a graduated cylinder for a known length of time.

5. Calculations

From the measured time interval and the wet test meter readings, the gas rate is converted to standard cubic feet per hour (SCFH at 29.92 in Hg and 60° F.).

The pounds per hour of hydrocarbon vapor lost to the atmosphere from the vapor leak is found by the following equation:

$$\text{pounds per hour} = \frac{(\text{SCFH})\ (\text{Mol. Wt.})}{379}$$

The volume of collected liquid is converted to a weight basis and added to the weight of vapor obtained from the above equation to obtain a total leakage in pounds of hydrocarbon per unit time.

SAMPLING OPERATIONS AND RESULTS

Because of the large number of valves and flanges in a refinery, and the number of refineries in the County, it is impractical to test every valve and flange for leakage.

Statisticians of the Department of Public Health, State of California, working with Mr. Hass, prepared a report which outlined a statistical approach to the sampling of valves and flanges in Los Angeles County refineries. Following the general approach suggested in their report, it was decided to divide each refinery into approximately 11 areas, each area representing a particular type of process. Three areas are to be sampled at each refinery, care being taken to ensure that each type of process is sampled an equal number of times.

In each area to be sampled, it was planned to test every sixth valve and flange, including those under 1-1/2 inches in diameter, and from this data to calculate to total leakage from this source. As the program got under way, however, it was found to be almost as easy to test every valve and flange for leakage, using a soap solution, as to count each one, so it was decided to test each valve and flange for leakage and to measure the leakage rate from every sixth one found to be leaking. Statistically, this method would yield more accurate data with very little more expenditure of effort.

To determine the extent of testing required, information was requested from the major oil companies and large independent oil companies on the number of valves and flanges in their refineries.

The results of this survey showed that there were:

1. About 56,000 valves and 76,000 flanges 1-1/2 inches in diameter and greater in service for products with volatilities as high or higher than gasoline.

2. About 19,000 valves and 30,000 flanges 1-1/2 inches in diameter and greater in service for products with volatilities in the range of kerosene.

About 90 per cent of the total valves and flanges are in the major oil companies.

Three field surveys were conducted to develop and test the procedure described. A refinery area (LPG Storage, Alkylation Plant, etc.) was chosen to be surveyed. The leaking fittings in the area were found by observation and spraying with a soap solution. It was noticed that the degree of leaking could be estimated by observing the rate of production of bubbles and measurements were made on those fittings estimated to have high leakage rates.

Since the surveys were made merely to develop and check the procedure, it was decided not to include the results in the data used for evaluation.

Four other surveys, using a revised procedure, were made in May, 1956. The results are summarized below:

Table IX

Valve Survey Results

Type of Service	No. of Valves Counted	No. of Valves Tested	No. of Leaks Found	Rate of Leakage lbs. per day (Estimates included)
Fractionating Unit	336	56	2	0.3
LPG	114	19	4	3.5
Gasoline or Lighter	522	87	21	160.3
LPG	1392	232	15	79.7
Total	2364	394	42	244

In survey No. 1, 56 valves in a fractionating unit were tested. Two small leaks, each estimated to be less than 0.1 CFH, were found.

In survey No. 2, 19 valves in LPG service were tested. Three leaks, each estimated to be less than 0.1 CFH, and one estimated to be 1.0 CFH were found.

In survey No. 3, 87 valves in gasoline or lighter service were tested. 21 leaks were found, of which three were measured and five were estimated to be over 1.0 CFH.

In survey No. 4, 232 valves in LPG service were tested. 15 leaks were found, of which 10 could not be measured, and three were estimated to be less than 0.1 CFH each. Two were measured and found to be leaking at the rates of 6.7 CFH and 18.2 CFH, the largest leaks measured thus far in the survey.

Estimates of leak rates were made where it appeared that the leak rate was less than 0.1 CFH or when time limitations or some other factor prevented actual measuring of the leak.

These four surveys were made by testing every sixth valve for leakage instead of testing every valve and measuring every sixth one found to be leaking.

Of 129 flanges tested, only one was found to be leaking and that was a blind flange. Measuring the leak proved unsuccessful because of faulty technique. This small percentage of flanges found leaking would seem to indicate that their importance, as sources of hydrocarbon leakage, is slight. It will be necessary to examine a greater number of flanges before this conclusion can be definitely established.

AIR BLOWING

1. Sampling Procedure
2. Sampling Operations and Results

IN BRIEF:

Air blowing is a minor operation performed in only a few refineries for brightening (removal of turbidity due to moisture) and for agitation during treating. The effluent air from these operations contains hydrocarbon vapor and aerosol.

The total losses to the atmosphere from air blowing operations are relatively small; however, the losses from the individual operations are such as to warrant consideration. For example, losses as high as 2 tons per day have been reported from one company.

A test procedure was developed and used several times but proved unreliable when aerosols were present in the sample. This has necessitated a revision to the procedure. Tests of two units show losses of 2,840 and 874 pounds per day. As a result of this testing program, one company has discontinued air blowing as a means of brightening.

SAMPLING PROCEDURE
for
AIR BLOWING OPERATIONS

1. Scope

This method describes a procedure for the determination of hydrocarbon losses from refinery operations in which air is blown through petroleum products for brightening or agitation.

2. Outline of Method

The gaseous effluent from the process vessel is collected in an evacuated sampling bulb. The concentration of hydrocarbons in the sample collected is measured by infrared spectrophotometry.

3. Apparatus and Materials

a. Thermometer

b. Pitot Tube

c. Inclined Draft Gauge

d. Evacuated 250 ml. Mine Sampling Bulbs

4. Sampling

A sampling hole suitably located for use in determining the effluent velocity is provided in the discharge stack of the process vessel. A Pitot tube traverse is taken and the rate of discharge is calculated (SCFM).

The process cycle is ascertained. If the process is continuous, the concentration of hydrocarbons in the effluent air stream will be constant.

If a batch operation is being tested, a number of samples must be taken to assess the variation of concentration with time.

The evacuated Mine Sampling Bulb inlet is placed in the effluent air stream and slowly opened. Temperature is recorded after the sample is taken. The samples are analyzed by Infrared Spectrophotometry.

5. Calculations

$$\text{Pounds per day of hydrocarbon vapor} = \frac{\text{ppm} \times \text{SCFM} \times \text{Mol. Wt.} \times 60 \times 24}{10^6 \times 379}$$

The concentration, in ppm (parts per million), is obtained from the Infrared analysis.

The rate of gas discharge, in SCFM (standard cubic feet per minute), is obtained from the velocity traverse (corrected for temperature).

The molecular weight (Mol. Wt.) is the average molecular weight of the petroleum fraction being processed.

SAMPLING OPERATIONS AND RESULTS

Air blowing is a minor operation and one which is not performed by all refineries. The air blowing operations considered are only those done in the agitating and brightening, or clarifying, of kerosene, diesel oil, stove oil and similar type stocks. The operation consists of introducing air into the bottom of the vessel containing the petroleum product. The air bubbles up through the liquid and is discharged from the vapor space of the vessel to the atmosphere.

Appropriate personnel of all of the refineries in the Los Angeles Basin were personally contacted and four items of information were requested for each air blowing operation except for asphalt air blowing. These items were:

1. The type of material being air blown.
2. The air blowing rate in cubic feet per hour.
3. The air blowing cycle.
4. The equipment arrangement and accessibility for sampling.

Air blowing operations were found at only three of the major oil companies and one of the smaller independent companies. The material being air blown was in general of a stove oil, diesel oil, jet fuel or kerosene type. Because of the small number of such air blowing installations in the County, it was proposed to test each operation. Since the survey was made, one of the major oil companies has reported that it has discontinued one of its air blowing operations and replaced it with settling and electrical separation methods.

Three field tests were conducted to develop and check the sampling procedure and analytical method. Subsequently, two additional tests were made with the results as shown in Table X.

Table X

Air Blowing Test Results

Test No.	Air Rate SCFM	Hydrocarbon Concentration ppm (as Hexane)	Pounds per day (as Hexane)
1	2,200	2,323	2,840
2	1,225	1,283	874

Since the above values were determined by an infrared spectrophotometer, calibrated for hexane, the actual values may be twice as large as shown.

The hydrocarbon emission rate in pounds per day was obtained from the following equation:

$$\text{pounds per day} = \frac{\text{ppm x SCFM x 180 x 60 x 24}}{10^6 \text{ x 379}}$$

The inadequacies of the infrared spectrophotometric method led to the consideration of other sampling methods:

1. By means of an air driven vacuum pump, the effluent is drawn through a series of three graduated freeze-out traps immersed in Dewar flasks containing a dry ice and petroleum thinner slurry. The remaining non-condensible material is then drawn through a wet test meter to measure the sample volume.

After sufficient sample is obtained, the traps are taken from the dry ice slurry and allowed to warm to atmospheric temperature. The water and oil that is collected forms two phases and the amount of oil in each trap is noted. It was stated by the company submitting this method that a fourth trap is not necessary. A combustible gas indicator may be used to determine whether any hydrocarbons are left in the exit stream.

2. The effluent is drawn through n-heptane. The change in the refractive index of the resulting solution is used to determine the concentration of hydrocarbons in the air stream. This method would not indicate any n-heptane collected.

3. The samples are collected in evacuated Mine Sampling Tubes and the concentration of hydrocarbons is determined by means of a Burrell-Haldane Gas Analysis Apparatus.

The Burrell-Haldane apparatus consists of a gas burette, a carbon dioxide absorption pipette, an oxygen absorption pipette and a catalytic combustion cell for the oxidation of hydrocarbons. A sensitive compensator manometer is employed and duplicate analyses can be checked to 0.02 %.

It is not necessary to know the composition of hydrocarbon samples since the determination is based on the measurement of CO_2 produced by combustion. The weight of hydrocarbon is calculated, assuming an average carbon content of 83%. Any aerosol deposited on the walls of the Mine Sampling Tube will be extracted with methylene chloride and analyzed by infrared spectrophotometry, using a liquid cell.

STORAGE TANKS

1. Calculation Method
2. Discussion and Results

IN BRIEF:

Hydrocarbon vapor losses from storage tanks have been calculated by methods presented at the 32nd Annual Meeting of the American Petroleum Institute in a Symposium entitled, "Evaporation Loss of Petroleum from Storage Tanks".

To obtain the data necessary for these calculations, surveys were made covering all refinery hydrocarbon tankage. A copy of the form used in this survey is in the Appendix, page xxxiv.

The results of these calculations are 15 tons per day of hydrocarbon losses from crude oil tanks and 27 tons per day from light petroleum distillate tanks, for a total of 42 tons per day.

CALCULATION METHOD
for
HYDROCARBON VAPOR LOSSES FROM STORAGE TANKS

Many methods for calculating hydrocarbon losses from storage tanks have been developed by various groups from experimental studies. The equations and nomographs used in this calculation method are from a Symposium presented at an American Petroleum Institute Annual Meeting.[7] It is not an official method of the American Petroleum Institute which has, however, formed a committee to prepare an "Evaporation Loss Manual."

The equations used for making the calculations are as follows:

1. Breathing Losses from Fixed Roof Tanks

$$B = \frac{PD^{1.8}}{14.5} F_o F_p$$

2. Filling Losses from Fixed Roof Tanks

$$F = \frac{3PV}{10,000} K_f$$

3. Evaporation Losses from Floating Roof Tanks

$$E = C\ P\ D$$

4. Filling Losses from Floating Roof Tanks

$$F_e = \frac{V}{10,000}$$

---

[7] "Evaporation Loss of Petroleum from Storage Tanks," 32nd Annual Meeting, American Petroleum Institute, November, 1952.

Where symbols used in the equations are:

B = breathing loss from a fixed roof tank, in barrels per year.

F = filling loss from a fixed roof tank, in barrels per year.

E = evaporation loss from a floating roof tank, in barrels per year.

F_e = filling loss from a floating roof tank, in barrels per year.

P = true vapor pressure at the average liquid body temperature, in pounds per square inch absolute.

D = tank diameter, in feet.

V = volume of liquid product added to the tank, in barrels per year (in the case where product is being withdrawn simultaneously with product addition, V is the excess of addition over withdrawal).

F_o = outage factor for fixed roof tanks.

F_p = paint factor for fixed roof tanks.

K_f = throughput factor.

C = floating roof factor for type of shell construction, seal, and roof.

Values to be used for the outage, paint, throughput and floating roof factors are:

1. Outage Factor, F_o

$$F_o = \frac{1}{5.7}(H + 0.5)^{0.57} - 0.1$$

Where H = outage, or height between top angle of the tank and, the liquid surface, in feet.

A table of outages versus outage factors prepared from the equation is as follows:

H	F_o
1	0.39
5	0.55
10	0.72
15	0.87
20	1.00
25	1.12
30	1.23
35	1.33
40	1.43
45	1.53
50	1.62

Some adjustments may have to be made to the outage value used if the volume under the tank roof is much different than the volume under a conical roof with a pitch of 3/4 inch per foot. A larger volume will call for a higher outage and a lower volume a lower outage.

2. Paint Factor, F_p

Paint	F_p
Chalking white	0.75
Aluminum	1.00
Light gray	1.10
Black, no paint or needs repainting	1.25

3. Throughput Factor, K_f

Tank Turnovers per year	K_f for General Refinery Service	K_f for Marketing Plants and Terminals
0 - 10	1.00	1.00
12	0.91	1.00
15	0.75	1.00
20	0.59	1.00
25	0.50	1.00
30	0.47	1.00
40	0.44	1.00
40 - 60		0.80
60-100		0.50

4. Floating Roof Factor, C

Shell Construction	Seal	C
Welded	Single or Double	0.49
Riveted	Double	0.56
Riveted	Single	1.20

Nomographs to be used in place of breathing and filling loss equations for fixed roof tanks are contained in the Symposium report. Correlations of Reid vapor pressures versus true vapor pressures for gasolines and crude oils are also presented in that report.

DISCUSSION AND RESULTS

The data required for making calculations of hydrocarbon vapor losses from petroleum tanks were obtained from each refinery in Los Angeles County. A copy of the data sheet used is in the Appendix, page xxxiv. Calculations were made as shown on the preceding pages.

The vapors lost to atmosphere were considered to have a density of 210 pounds per barrel in the liquid state.

Tanks connected to vapor recovery systems were assumed to have no losses.

The equations and nomographs used for estimating losses from fixed roof tanks are based on the following assumptions:

1. Roofs are vapor-tight.
2. Vents, gauge hatches and other accessories are vapor-tight.
3. Tanks operate at essentially atmospheric pressure; i.e., with vent pressure and vacuum settings in the order of 0.5 ounce per square inch.
4. The volume under the roof is essentially the same as the volume under a low pitch conical roof having a slope of 3/4 inch per foot.
5. The mean daily temperature change is 16° F.

The equations used for estimating losses from floating roof tanks are based on the following assumptions:

1. Average wind velocities are 5 to 10 miles per hour.

2. Tanks are in good condition.
3. Tank shells and roofs are painted aluminum or gray.
4. Floating roofs and sealing rings are of the type which were in service 8 to 20 years ago.

The losses predicted from floating roof tanks are probably high. This is because many of the floating roof tanks in Los Angeles County have newer type seals and roofs, are painted white and experience lower average wind velocities than were assumed in the derivation of the equation.

Calculated hydrocarbon vapor losses from crude oil tanks are 15 tons per day and from light petroleum distillate tanks are 27 tons per day, a total of 42 tons per day.

Detailed results of the surveys and hydrocarbon loss calculations are presented in Tables XI and XII.

Table XI

Crude Oil Tanks at Los Angeles County Refineries

Size Range of Tanks	Type of Tank	Vapor Pressure Below 1.5 PSIA						Vapor Pressure Above 1.5 PSIA					
		Number		Capacity		Losses		Number		Capacity		Losses	
		No.	%	Barrels	%	lbs/day	%	No.	%	Barrels	%	lbs/day	%
Less than 1,000 bbls.	Fixed Roof	1	0.7	500	0.0	0	0	1	0.7	750	0.0	20	0.0
	Floating Roof							1	0.7	836	0.0	34	0.1
	Vapor Recovery							1	0.7	500	0.0	0	0.0
1,000 to 24,999 bbls.	Fixed Roof	16	11.9	157,587	2.1	1,053	3.5	32	23.7	140,553	1.4	4,322	14.5
	Floating Roof												
	Vapor Recovery												
25,000 to 49,999 bbls.	Fixed Roof	3	2.2	115,421	1.6	600	2.0	2	1.5	74,000	1.0	1,246	4.2
	Floating Roof							1	0.7	49,852	0.7	174	0.6
	Vapor Recovery												
50,000 to 74,999 bbls.	Fixed Roof	1	0.7	53,886	0.7	18	0.0	7	5.2	386,520	5.2	1,352	4.6
	Floating Roof							4	3.0	220,259	3.0	1,096	3.7
	Vapor Recovery												
75,000 to 99,999 bbls.	Fixed Roof	1	0.7	80,000	1.1	135	0.5	4	3.0	320,24[illegible]	4.3	3,015	10.1
	Floating Roof							4	3.0	307,431	4.2	1,223	4.1
	Vapor Recovery	2	1.5	152,228	2.1	0	0.0	30	22.2	2,354,638	31.9	0	0.0
100,000 bbls. and larger	Fixed Roof	4	3.0	400,000	5.4	1,782	6.0	8	5.9	986,199	13.4	7,462	25.1
	Floating Roof	4	3.0	536,000	7.3	942	3.2	8	5.9	1,045,497	14.1	5,291	17.8
	Vapor Recovery												
TOTAL	Fixed Roof	26	19.2	807,400	10.9	3,588	12.0	54	40.0	1,908,263	25.8	17,420	58.4
	Floating Roof	4	3.0	536,000	7.3	942	3.2	18	13.3	1,623,875	22.0	7,818	26.3
	Vapor Recovery	2	1.5	152,228	2.1	0	0.0	31	23.0	2,355,138	31.9	0	0.0
		32	23.7	1,495,628	20.3	4,530	15.2	103	76.3	5,887,276	79.7	25,238	84.8

Table XII

Light Petroleum Distillate Tanks at Los Angeles County Refineries

224 Pressure Tanks
713,350 bbls. Capacity

Size Range of Tanks	Type of Tank	Vapor Pressure Below 1.5 PSIA						Vapor Pressure Above 1.5 PSIA					
		Number		Capacity		Losses		Number		Capacity		Losses	
		No.	%	Barrels	%	lbs/day	%	No.	%	Barrels	%	lbs/day	%
Less than 1,000 bbls.	Fixed Roof	20	2.8	6,815	0	77	0.1	25	3.6	12,608	0	592	1.1
	Floating Roof	5	0.7	2,256	0	10	0	4	0.6	1,840	0	140	0.3
	Vapor Recovery	3	0.4	1,878	0.1	0	0	18	2.6	7,044	0	0	0
1,000 to 24,999 bbls.	Fixed Roof	40	5.7	133,398	0.6	380	0.7	*6	0.9	*70,000	0.3	*95	0.0
	Floating Roof	57	8.1	483,989	2.3	1,620	3.0	92	13.1	994,264	4.8	8,132	14.8
	Vapor Recovery	23	3.3	172,761	0.8	0	0	166	23.6	756,570	3.6	0	0
25,000 to 49,999 bbls.	Fixed Roof	2	0.3	64,705	0.3	137	0.3						
	Floating Roof	12	1.7	464,705	2.2	604	1.1	33	4.7	1,114,120	5.4	6,586	12.
	Vapor Recovery	2	0.3	60,000	0.3	0	0	4	0.6	146,210	0	0	0
50,000 to 74,999 bbls.	Fixed Roof	3	0.4	165,929	0.8	563	1.0						
	Floating Roof	15	2.1	876,554	4.2	1,344	2.5	27	3.8	1,631,245	7.9	8,793	16.
	Vapor Recovery							3	0.4	177,108	0	0	0
75,000 to 99,999 bbls.	Fixed Roof	5	0.7	401,189	1.9	401	0.7						
	Floating Roof	5	0.7	407,336	2.0	681	1.2	42	6.0	3,376,289	16.2	14,638	26.7
	Vapor Recovery	4	0.6	320,346	1.5	0	0	40	5.7	3,132,764	15.1	0	0
100,000 bbls. and larger	Fixed Roof	2	0.3	217,253	1.0	20	0.0	*4	0.6	*536,000	2.6	*705	1.3
	Floating Roof	7	1.0	893,789	4.3	1,006	1.8	21	3.0	2,680,263	12.9	8,368	15.3
	Vapor Recovery							14	2.0	1,496,031	7.2	0	0
TOTAL	Fixed Roof	72	10.2	991,289	4.8	1,578	2.9	35	5.0	618,608	3.0	1,392	2.5
	Floating Roof	101	14.3	3,128,629	15.0	5,265	9.6	219	31.1	9,798,021	47.0	46,657	85.5
	Vapor Recovery	32	4.5	554,985	2.7	0	0	245	34.8	5,715,727	27.4	0	0
		205	29.1	4,674,903	22.5	6,843	12.4	499	70.9	16,132,356	77.5	48,049	87.6

* Vapor balance

PUMPS

1. Sampling Procedure
2. Testing Program

IN BRIEF:

Pumps can be a source of hydrocarbon emissions to the atmosphere because of leakage through seals and packing glands.

A procedure has been prepared for measuring pump leakage based on work done previously by the industry. A field testing program for pumps is to be undertaken after requisite information on the pumps in refineries has been obtained.

SAMPLING PROCEDURE
for
PUMPS

1. Scope

This method describes a procedure for the determination of hydrocarbon losses to the atmosphere from pump seals and packing glands. Both vapor and liquid hydrocarbon leakages are considered.

2. Outline of Method

The suspected leak area is enclosed in a shroud not necessarily airtight. Hydrocarbon vapors from the leak are drawn with a controlled amount of air through a meter by use of a vacuum pump or aspirator for a measured length of time. The hydrocarbon concentration in the mixture is determined by a combustible gas indicator.

Liquid from the pump seals or packing is collected and the hydrocarbon content determined. The fraction of the liquid which would have been lost to the atmosphere under prevailing conditions is estimated and added to the vapor loss.

3. Apparatus and Materials

a. Vacuum pump (explosion-proof electric or air drive) or Aspirator (air, steam or water ejector)

b. Surge bottle

c. Combustible Gas Indicator

d. Wet Test Meter

e. Rotameter (optional)

f. Stopwatch

g. Graduated cylinder (25 ml.) and Funnel

h. Tygon tubing and Screw clamps

i. Polyethylene or Vinyl plastic sheeting

j. Masking tape

k. Soap solution and Brush or sprayer

DIAGRAM OF PUMP LEAKAGE TESTING EQUIPMENT

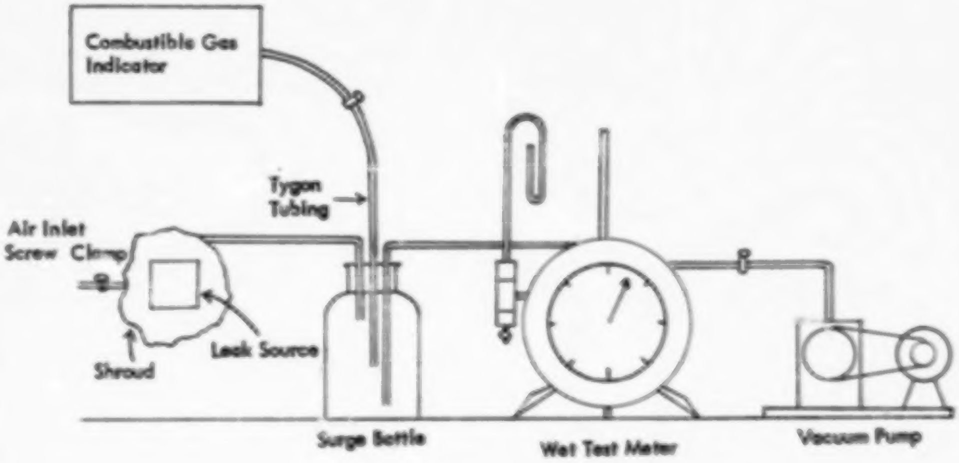
Combustible Gas Indicator
Tygon Tubing
Air Inlet Screw Clamp
Leak Source
Shroud
Surge Bottle
Wet Test Meter
Vacuum Pump

Figure 12

4. Assembly of Apparatus

The vapor sampling equipment is arranged as shown in Fig. 12. The polyethylene or vinyl plastic sheet is wrapped around the leak source as a shroud and sealed with masking tape. A wire frame may sometimes be needed to hold the shroud away from the shaft. The air inlet to the shroud should be so located that the air will sweep past the hydrocarbon vapor leak. During the test a negative pressure should be maintained in the shroud.

A rotameter may be placed in the line, before the wet test meter, to provide a rapid means of checking the constancy of flow rate.

5. Sampling

The system is purged by the vacuum pump for about 10 minutes at a rate which maintains a slight vacuum in the shroud. After steady flow conditions are obtained, the rate is determined by timing the wet test meter. The hydrocarbon content is checked with the combustible gas indicator. It may be necessary to change the flow rate in order to bring the hydrocarbon concentration within the range of the instrument. When an acceptable flow rate is obtained, several determinations of the hydrocarbon concentrations are made and the average calculated.

Samples of total liquid drippage are collected in bottles for a convenient time interval. The hydrocarbon content is measured volumetrically.

6. Calculations

From the measured time interval and the wet test meter readings, the gas flow rate is converted to standard cubic feet per hour (SCFH).

The volume percentage of the appropriate hydrocarbon in the gas is obtained from the combustible gas indicator readings and the L. E. L. conversion curves. (See Fig. 8.)

The pounds per hour of hydrocarbon vapor lost to the atmosphere from the vapor leak is found by the following equation:

$$\text{pounds per hour of hydrocarbon vapor} = \frac{(\text{SCFH})(\text{vol.\% hydrocarbon})(\text{mol.wt.})}{(100)\ (379)}$$

If liquid hydrocarbon leakage is found, an estimate is made of the amount which would evaporate under the specific conditions encountered.

TESTING PROGRAM

Letters have been sent to the refineries requesting information as to the number of pumps in use, products handled, types of seals, operating temperatures and operating service.

This information will be used to develop a testing program.

CATALYTIC CRACKING UNIT
REGENERATOR STACKS

1. Sampling Procedure
2. Testing Program

IN BRIEF:

The refineries in Los Angeles County operate six Fluid Catalytic Cracking Units, nine Thermofor Catalytic Cracking Units, and one Thermofor Catalytic Reforming Unit.

Thirteen tests are to be conducted to measure the emissions of particulate matter, aldehydes, hydrocarbons, ammonia, oxides of sulfur, and other contaminants.

SAMPLING PROCEDURE
for
CATALYTIC CRACKING UNIT REGENERATOR STACKS

1. Scope

This method describes a procedure to be used in testing catalytic cracking unit regenerator stack gases for:

Aldehydes
Ammonia
Carbon monoxide, carbon dioxide
  and oxygen
Cyanides
Hydrocarbons
Organic acids
Oxides of nitrogen
Oxides of sulfur
Total particulate matter
Total volume of stack gas and
  per cent moisture

2. Outline of Method

Sampling for particulate matter, ammonia, oxides of sulfur, organic acids and cyanides is carried on simultaneously and continuously for a period of one hour with two parallel sampling trains. A gas sample for Orsat analysis is collected by a liquid displacement method. During the sampling period, grab samples for aldehydes, oxides of nitrogen and hydrocarbons are taken.

3. Apparatus and Materials

a. Glass probes (Vycor)
b. Whatman thimble, single thickness 43mm. x 123mm.
c. Whatman thimble, double weight 43mm. x 123mm.
d. Alundum thimble--porosity RA 98 coarse, 45mm. x 127mm.
e. Vacuum pump, or air aspirator
f. Six 500 ml. impingers.
g. Integrating type dry meter

h. Mercury manometer and thermometer
i. Evacuated bulbs--300 ml. Mine Sampling Tubes
   and round bottom 2 liter flasks
j. Pitot tube and inclined draft gauge
k. Whatman and Alundum thimble holders
l. Ice bath container
m. Thermocouple pyrometer

4. Sampling

A convenient stack sampling point is chosen in a region of uniform flow. The velocity of the effluent stream is measured by making a Pitot tube traverse. Samples are withdrawn continuously from a point of average velocity, maintaining isokinetic velocities at the two sampling nozzles.

a. The two trains following the sampling probes are:

   (1) An Alundum thimble (with a pyrex glass wool lining) followed by three 500 ml. impingers immersed in an ice bath. The first impinger contains 100 ml. of distilled water. The second impinger contains 100 ml. of a 5% solution of HCl. The last impinger is dry and is provided with a thermometer and manometer. The impinger section is followed by a meter and vacuum pump.

   (2) A double thickness Whatman paper thimble followed by three 500 ml. impingers immersed in an ice bath. The first two impingers each contain 100 ml. of a 5% solution of NaOH. The third impinger is dry and is provided with a thermometer. Following the impinger section are a single thickness Whatman paper thimble, meter and vacuum pump.

b. An integrated gas sample for Orsat analysis is collected by withdrawing gases continuously from the stack at a constant rate by a liquid displacement method. A five liter bottle filled with a saturated sodium sulfate solution, acidified with sulfuric acid, is used as the gas holder. As the solution siphons out, the gas sample is drawn into the bottle. The rate of siphoning is adjusted so that a gas sample of about four liters is obtained during the course of the test. The sample is drawn from the same cross section used for collecting the particulate matter.

c. During the sampling period, four grab samples are taken at the end of every 15 minute interval as follows:

  (1) Aldehydes: A gas sample is drawn into a 2-liter evacuated flask containing 10 ml. of a 1% solution of sodium bisulfite.

  (2) Oxides of Nitrogen: A gas sample is drawn into a 2-liter evacuated flask containing 25 ml. of a solution of hydrogen peroxide and sulfuric acid. The absorbing solution is prepared by adding 1 ml. of 3% H_20_2 to 100 ml. of 0.1N H_2S0_4.

  (3) Hydrocarbons: Two gas samples are taken. One sample is collected in a dry evacuated 2-liter flask. This sample is analyzed on an Infrared Spectrophotometer and reported as hexane. The second sample is collected in a 300 ml. dry evacuated Mine Sampling Tube. This sample is analyzed by a Mass Spectrometer.

5. Analyses (See Appendix for detailed procedures)

   a. Alundum Thimble: The particulate matter in the thimble is weighed and ignited at 1200°F. Weight of particulate matter collected and loss of weight on ignition are reported.

   b. Impinger train (Water-HCl): The increase in volume of the impinger solutions is measured and knowing the temperature and pressure of the saturated gas in the dry impinger, the per cent moisture in the stack gas is determined. The impinger solutions are filtered separately and the residues weighed. An aliquot of the combined, filtered impinger solutions is made alkaline with sodium hydroxide and distilled into a standard H_2SO_4 solution, which is then back titrated using a standard sodium hydroxide solution, for the determination of ammonia.

   c. Double weight Whatman paper thimble: The SO_3 is extracted with water in a Soxhlet Extraction Unit and determined gravimetrically.

   d. Impinger train (5% NaOH): The impinger solution is used for three determinations:

      (1) Total Oxides of Sulfur: An aliquot is oxidized and the sulfur oxides are determined gravimetrically.

      (2) Organic Acids: An aliquot is acidified and ether extracted. The organic acids in the ether layer are titrated with standard sodium hydroxide solution.

(3) Cyanides: An aliquot is treated with acid, steam distilled into a sodium carbonate solution and the solution reacted with picric acid. The concentration is determined colorimetrically.

e. Grab Samples:

(1) Aldehydes: The sodium bisulfite-aldehyde complex is determined by an iodine titration.

(2) Oxides of Nitrogen: The absorbing solution is made alkaline and evaporated to dryness. Phenoldisulfonic acid solution is added to the residue and the determination of the total oxides of nitrogen is made by measuring the light absorption of the resulting yellow solution, in a photoelectric colorimeter.

(3) Hydrocarbons: The gas sample collected in the 2-liter dry evacuated flask is purged into an Infrared Spectrophotometer and analyzed for total hydrocarbons as hexane.

The gas sample collected in the 300 ml. dry evacuated Mine Sampling Tube is analyzed in a Mass Spectrometer for the following compounds:

Methane
Ethane
Propane
Ethene
Propene
mixed Butanes
mixed Pentanes
mixed Hexanes
mixed Heptanes
Aromatics
total Cycloparaffins and Olefins

TESTING PROGRAM

Arrangements are being made to assign personnel from the APCD Research Division to a Joint Project testing team to carry out the Catalytic Cracking Unit testing program.

The six Fluid Catalytic Cracking Units, six of the nine Thermofor Catalytic Cracking Units, and the one Thermofor Catalytic Reforming Unit are to be tested.

APPENDIX

1. Copy of letter, dated August 4, 1955, from S. Smith Griswold, Air Pollution Control Officer, to Board of Supervisors, Los Angeles County, recommending participation in a Joint Project.............................. ii

2. Board Order, dated August 9, 1955, authorizing Air Pollution Control District participation......................... v

3. TABLE XIII--List of Analytical Methods for Stack Gases........ vii

4. TABLE XIV--Summary of Los Angeles County Refinery Testing Procedures viii

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    Organic Acids.. xxx
    Oxides of Nitrogen................................... xxxi
    Oxides of Sulfur..................................... xxxiii

6. Storage Tank Data Sheet................................ xxxiv

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August 4, 1955

Honorable Board of Supervisors
County of Los Angeles
501 Hall of Records

Gentlemen:

Subject: Federal and State Participation with the Air Pollution Control District in the Reevaluation of Air Contaminants from Refineries and Power Plants and the Air Monitoring Program in the Los Angeles Basin.

Recent recognition by the Federal Government of its air pollution control responsibilities resulted in meetings to assist in program developments. Although $25,000,000 has been authorized by Congress, none has been appropriated, and the Federal agency is now organizing and operating under a limited emergency appropriation. However, Los Angeles County will immediately benefit.

The Air Pollution Control Officer met with representatives of the Federal and State air pollution control agencies on July 22, 25, and 27. These meetings were attended by Dr. Louis C. McCabe, Dr. Arthur C. Stern, and Dr. Vernon McKenzie of the Federal air pollution control agency, and Dr. Malcolm Merrill and Dr. Frank M. Stead of the California Department of Public Health.

As a result of these meetings, it was mutually agreed that the State and Federal governments would assign highly qualified technicians to work in the Air Pollution Control District organization for a period of six months beginning September 1, or immediately thereafter, on two specific projects:

1. A reevaluation of contaminants emitted into the air from refineries and power plants operating in the Los Angeles Basin. Preliminary work has already begun. The Air Pollution Control District is now developing effective sampling procedures to secure for laboratory analysis air samples taken from the visible plumes from refineries and power plants. Techniques have been discussed with the staff of technical consultants, Dr. Ulric B. Bray, Dr. A. J. Haagen-Smit, and Dr. Francis E. Blacet.

   Additional meetings are scheduled to finalize the techniques to be used prior to September 1, 1955.

2. The air monitoring program at 14 stations now being developed as a continuous operation in the Los Angeles Basin as required under Regulation VII. This project includes the evaluation of new automatic instruments now being developed under supervision of District technicians.

These projects are considered to be most important to the health and welfare of the people of Los Angeles and will be accomplished as quickly as possible.

The Federal and State governments agreed to maintain four technicians in this area at their own expense. It was also agreed that these personnel would work as a part of the operating staff of the District. The State and Federal agencies will, in addition, act in a consulting capacity in evaluating the data developed. The State and the Federal technicians assigned to the projects will be under the supervision of the Air Pollution Control District. The State and Federal agencies will participate in developing the findings and conclusions.

It was originally expected that the Federal agency would assign a greater number of technical personnel to the Los Angeles area. It is understandable, however, that greater participation by the Federal agency would be extremely difficult at the present time. The Federal agency is now in the process of being organized. The greater portion of their technical staff must be assigned to the procurement and evaluation of data existing in the field of air pollution. Recruitment of technical personnel with experience in air pollution research is extremely difficult.

It is expected that as the Federal government progresses in developing an effective organization, an increasing number of technicians will be assigned to air pollution research in the Los Angeles Basin.

The procedure established by the Federal government contemplates action through a state air pollution control agency where such exists. It was therefore agreed that the Los Angeles County Air Pollution Control District would formalize the understanding by submitting an application through the California State Department of Public Health.

In order to comply with this understanding, it is recommended that:

> The Board of Supervisors, acting as the Air Pollution Control Board of the County of Los Angeles, approve the proposals outlined herein, and
>
> instruct the Clerk of the Board to prepare and authorize the Chairman to sign an application to the State Department of Public Health which will implement the determinations described herein.

It is understood that a copy of the Board Order, presented as an application properly signed by the Chairman, will suffice.

Yours very truly,

S. Smith Griswold
Air Pollution Control Officer

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Y

COUNTY OF LOS ANGELES
Board of Supervisors
501 Hall of Records
Los Angeles 12

Members of the Board

Herbert C. Legg
Chairman
Kenneth Hahn
John Anson Ford
Burton W. Chace
Roger W. Jessup

TUESDAY, AUGUST 9, 1955

The Air Pollution Control Board of the Air Pollution Control District met in regular session. Present: Supervisors Herbert C. Legg, Chairman presiding, Kenneth Hahn, John Anson Ford, and Burton W. Chace; and Harold J. Ostly, Clerk, by John R. Leach, Deputy Clerk. Absent: Supervisor Roger W. Jessup.

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IN RE FEDERAL AND STATE PARTICIPATION WITH THE AIR POLLUTION CONTROL DISTRICT IN THE REEVALUATION OF AIR CONTAMINANTS FROM REFINERIES AND POWER PLANTS AND THE AIR MONITORING PROGRAM IN THE LOS ANGELES BASIN: ORDER APPROVING RECOMMENDATIONS OF THE AIR POLLUTION CONTROL OFFICER, AND INSTRUCTING CLERK TO PREPARE AND CHAIRMAN TO SIGN APPLICATION TO STATE DEPARTMENT OF PUBLIC HEALTH.

A communication signed by the Air Pollution Control Officer, dated August 4, 1955, stating that recent recognition by the Federal government of its air pollution control responsibilities resulted in meetings to assist in program developments and that although $25,000,000 has been authorized by Congress, no money has been appropriated; and as a result of these meetings it was mutually agreed that the State and Federal governments would assign highly qualified technicians to work in the Air Pollution Control District organization for a period of six months beginning September 1, 1955, or immediately thereafter on the following projects:

1. A reevaluation of contaminants emitted into the air from refineries and power plants operating in the Los Angeles Basin.
2. An air monitoring program at 14 stations as a continuous operation in the Los Angeles Basin as required under Regulation VII (this project including the evaluation of new automatic instruments now being developed under supervision of District technicians);

the Federal and State governments agreeing to maintain four technicians in this area at their own expense and that State and Federal personnel will work as a part of the operating staff of the District; the State and Federal agencies, in addition, to act in a consulting capacity in evaluating the data developed; the State and Federal technicians assigned to the projects to be under the supervision of the Air Pollution Control District and the State and Federal agencies to participate in developing the findings and conclusions; and it being agreed that the Los Angeles County Air Pollution Control District will formalize the understanding by submitting an application through the California State Department of Public Health, - is presented; and on motion of Supervisor Hahn, unanimously carried, it is ordered that the proposals contained in said communication be and the same are hereby approved and that the Clerk of this Board be and he is hereby instructed to prepare and the Chairman of this Board be and he is hereby authorized and directed to sign in behalf of the Air Pollution Control District an application to the State Department of Public Health which will implement the determinations described in said communication.

It is further ordered that this order shall be deemed an application for Federal and State assistance in the Air Pollution Control Program as outlined above.

Herbert C. Legg, Chairman
Air Pollution Control Board of the
Air Pollution Control District of the
County of Los Angeles, State of California

TABLE XIII

List of Analytical Methods Used by Western Oil and Gas Association Members for Stack Gas Analysis

Substance Determined	Method Used	Comments
Aldehydes	API 762-54 or RCWD-13	Sample was collected in an evacuated bottle in the API method and passed through scrubbers in the RCWD method. Chemistry is same in both.
Ammonia	API 763-54	Colorimetric modification was used for low ammonia concentrations.
Carbon monoxide	Mine Safety Appliance CO tester or Orsat Analyzer	M. S. A. tester was used for low CO concentrations (furnace stacks), and the orsat tester for high CO concentrations (regenerator stacks).
Hydrocarbons	Mass spectrometer or IR absorption methods on "as obtained" or on concentrated samples	M. S. limited to above 50 ppm hydrocarbons. For low concentrations, two companies employed the freezeout technique to concentrate hydrocarbons. Results by this method were not too reliable.
Organic acids	No standard method	Technique used by one member: Absorption in caustic, followed by removal of interfering inorganic acids and titration.
Oxides of nitrogen	Standard APCD phenoldisulfonic acid method	Total oxides of nitrogen were determined. Total oxides of nitrogen include NO_2 and NO.
Oxides of sulfur	API 774-54	Total oxides of sulfur were determined. This method recommended when analysis for sulfur content of fuel being burned is not practical. When analysis for sulfur content of fuel is practical (fuel oil and fuel gas), recommend using ASTM D129-52 for liquid fuels and Tutweiler method for hydrogen sulfide content, the sulfur dioxide emissions can be calculated. API method 774-54 is not entirely reliable due to interference of other stack gas constitutents.

TABLE XIV

Summary of Los Angeles County Refinery Testing Procedures

	Subject	Company	Comments
1.	Cooling towers Hydrocarbons	A	Method of Hass and Sussman, APCD. 10-18-55 (J.D.F.S. Project).
		B	Only experience with recent work with J.D.F.S. Project.
		C	No comment.
		D	APCD Method, also a 500-ml. sample of return water into 1-liter evac. flask shaken to release HC vapor anal. by m.s. Losses shown insignificant.
2.	Air blowing of kerosene, diesel oil, etc. Hydrocarbons	A	No air blowing operations.
		B	No tests; losses estimated from vol. of air used assuming becomes satd. with HC being air blown.
		C	No air blowing operations of this kind.
		D	Air rate in by meter. Effluent drawn through 3 grad. freeze out traps in dry ice slurry. Non-condensibles meas. by w. test meter. Traps warmed up - oil separated as second phase and measured.
3.	Storage of heavy hydrocarbons at elevated temperatures Hydrocarbons	A	No experience.
		B	No experience.
		C	Suggest use of API Method of calculation, modified for no breathing loss.
		D	No comment.

Subject	Company	Comments
4. Sewer boxes, lines, sumps Hydrocarbons	A	Sewer boxes have traps. No escape of hydrocarbons detected.
	B	Losses estimated as an arbitrarily selected fraction of the separator box losses.
	C	Sewer box covered with plywood and gas escape meas. by anemometer and sampled for anal. by m. s. No sump info.
	D	Anemometer or pitot tube at effluent point. Sample taken in evac. flask for m. s. analysis for 100 ppm or greater of C_8 or ligher hydrocarbons.
5. Flanges, other pipe connections, and valves. Hydrocarbons	A	No losses. Flanges hydrostat. tested at press. higher than service. Any leak fixed immediately.
	B	No experience.
	C	No experience.
	D	Volumetric measure of liquid leaks. Periodic survey.
6. Pipeline blind changing Estimate hydrocarbon liquid and gas loss.	A	Estimates based on observations. Hamer blinds used where frequent change required. Losses based on number of blind changes per day.
	B	Liquid losses detd. for typical change. For vapor, grav. and v.p. of liquid escaping flange compared with liquid running into closed drains--difference determined for vapor loss.
	C	Estimated from known number of changes and determination of typical liquid loss. Vapor loss estimated.
	D	Estimate based on line size and length of line drained. If gaso. drains on slab assume 20% to air. If to ground 100% loss. LPG - all to air.

	Subject	Company	Comments
7.	Relief valves (accumulators, process vessels, etc.) Hydrocarbons	A	Accum. valves to vapor recovery or ride on compressor, or vented to flare, etc. Losses attributed to this source very low and are estimated.
		B	Estimated from total length of time during typical year valve forced open and the standard flow rate for each valve.
		C	To vapor recovery system.
		D	Where discharged to air loss estimated on pop frequency - negligible here.
8.	Loading stations for hot, heavy hydrocarbons (asphalt, fuel oils, etc.) Hydrocarbons	A	No experience.
		B	No experience.
		C	No comment.
		D	No comment.
9.	Loading stations for light hydrocarbons (including kero., jet fuel, solvents, gaso., etc.) Hydrocarbons	A	Loading tanks, trucks, tank cars, etc., follow same pattern. Freeze out samples for anal. taken of vapor space. Displacement and turbulence loss calcd.
		B	HC content of samples composited from gas escaping through hatches during typical loading. Vol. of vapors est. as vol. of liquid loaded plus vol. of HC vaporized.
		C	Tanker loading. O_2 determined in samples by Orsat and HC detd. by diff. Checked by absorbing HC in oil - also m. s. See also Nat. Pet. News p. 22, July 13, 1949.
		D	Anemometer or pitot tube at effluent point. Evac. flask sample for m. s. analysis.

	Subject	Company	Comments
10.	Waste water separators Hydrocarbons	A	Vapor pres. and grav. of influent and effluent oil as basis. New system enclosed. Air retention tanks and clarifiers to vapor recovery.
		B	Grav., B.P. of oil entering and leaving used. See Viles and Jones, Pet. Ref. 31, 117-19 (Jan. 1952) for method.
		C	Losses detd. from properties of in and outgoing oil. Confirmed by lab. evap. expts. No loss now detectable since covered. Zero explosion meter reading.
		D	See Method 10D.
11.	Storage of light hydrocarbons of v.p. 1.5 psia at storage conditions (includes crude oil tanks and tanks under 40,000 gal. capac.) Vents for hydrocarbons.	A	Calculated by API Method as presented at the 32nd Annual API Meeting, Nov. 10, 1952.
		B	Calculated by API Method.
		C	Calculated by API Method.
		D	Calc. by API Method. Includes all tanks that store prods. having measurable Reid v.p.
12.	Waste gas flares Hydrocarbons and smoke	A	No experience on tests. Pilot light is always burning. Smoking would be due to failure of steam control valves. Passes LAAPCD test.
		B	No experience.
		C	No comment.
		D	No comment.
13.	Blow down stacks Hydrocarbons	A	Operating units pumped out - gas to holder or recovery. Only blow down is small amt. gas oil from heater coils. Oil through coolers. Small loss estimated.
		B	Composited samples of stack gas were anal. for HC. The stack gas rate estim. using a metered flow of helium as tracer.
		C	Go to vapor recovery system.
		D	Recording pitot tube in stack for flow. Samples in evac. flask analyzed by ms.

	Subject	Company	Comments
14.	Vacuum jets Hydrocarbons	A	No experience.
		B	Composited samples of gas anal. for H.C. Volumes estim. using metered flow of nitrogen or helium as tracer.
		C	All jets now emit to incinerator or furnace. However, see Method 14-C.
		D	Evac. flask - m.s. analysis.
15.	Pumps in hydrocarbon service Hydrocarbons	A	See Method 15A.
		B	See Method 15A.
		C	No comment.
		D	Survey pumps - packing gland, oil seal, or mech. seal. Classify as to service; i.e., LPG, gasoline, or heavy gasoline. Estimate based on Method 15A for each classification.
16.	Acid sludge storage and handling (filling trucks and tank cars) Hydrocarbons, SO_2	A	During loading vent gases through scrubber. Small amts. escaping determ. by gas sampling tubes and m.s. Acid sludge loading will be eliminated early in 1956.
		B	Conc. of SO_2 in vapor space by Orsat for total acidics. Breathing and filling losses for SO_2-modification of API hydrocarbon method used.
		C	Method same as in Item 9C for tanker loading.
		D	See Method 16D
17.	Sour water strippers Mercaptans H_2S NH_3	A	RSH by RSAg. Excess $AgNO_3$ used and back tit. with NH_4SCN. $FeSO_4$ as indic. H_2S as PbS-weighed. Low conc. APHA, 10th Ed. 1955. NH_3 by dist. from alk. soln. into boric acid.
		B	Sulfur was estimated from a balance around the unit. H_2S and NH_3 burned in furnace.
		C	Gas from strippers goes to ammonium sulfate plant.
		D	See Method 17D.

	Subject	Company	Comments
18.	Air blown asphalt stills Hydrocarbons	A	Gas collected in sampling tubes,determined by mass spectrometer. Rate by pitot tube. HC content low. Total effluent to odor abatement furnace for disposal.
		B	No experience.
		C	Effluent is incinerated.
		D	Air in by meter. Effluent of plant scrubbed through salt water. No HC found in dry ice or liquid N_2 freeze out traps.
19.	Air agitators as in acid treating units Hydrocarbons SO_2	A	No such operation here.
		B	See Item 2B.
		C	No such operation now.
		D	Anemometer or pitot tube at effluent point. Method same as No. 2D.
20.	Doctor treating units Hydrocarbons	A	No tests made. Closed system under pressure.
		B	No experience.
		C	See Method 20C. HC loss now nil. New plumbite stripper installed.
		D	Air rate to unit measured by meter. Based on V.P. of stock being treated. Air discharged to atmos. assumed to contain equilibrium conc. of HC. Loss then calc.
21.	H_2S removal plant stacks SO_2	A	No experience. H_2S stripped from DEA solution and sent to sulfur recovery plant.
		B	No experience.
		C	H_2S streams go to sulfur plant for production of elemental sulfur. Final effluent gas incinerated SO_2 by KOH-H_20 method on incin. stack.
		D	No comment.

	Subject	Company	Comments
22.	Gasoline mercaptan removal plants	A	Removed mercaptans sent to odor abatement heater. Samples from reclaimer release analyzed by m. s.
	Hydrocarbons Mercaptans	B	All venting from this plant burned in furnace. Assumed all HC burned. Mercaptans estimated from sulfur balance.
		C	No loss to atmosphere.
		D	No comment.
23.	Process Unit shutdown for equipment inspections. Estimate hydrocarbon, liquid and gas losses.	A	Vessels reduced to atmos. pres.; steam pushes remaining vapor to gas holder. See Item 13A.
		B	No experience.
		C	Liquid to tankage. Gas to vapor recovery system. Small amount of low-spot line bleeding to sewer. No estimates.
		D	Determine by survey no. and size of tanks per yr. taken out of service. Determine which contained HC prior to gas freeing. Assume 100% HC content in vapor space and calc. loss.
24.	Catalyst emissions from TCC units during Rotoclone shutdown. Catalyst dust.	A	Only down about 4 hrs. in operating cycle. Normal dust test by modification of Western Precip. Corp. Bull. WP-50, 4th Edition, Gas & Dust Measurements.
		B	No experience.
		C	Steam control to TCC elevators prevents catalyst pipe clogging. Rotoclone not shut down during normal operation.
		D	No comment.
25.	Tank car cleaning	A	No experience.
	Hydrocarbons from solvents	B	No experience.
		C	No experience. Suggest use of explosion meter.
		D	No comment.

TABLE XIV

METHOD 10D

Hydrocarbons from Waste Water Separators

The method described was used for a multi-compartment type separator with the inlet compartments sealed with a vapor tight cover fitted with a breather valve used as a pressure relief. The balance of the compartments were covered with close fit wood planks.

To determine the losses occurring from the vapor tight compartments through the breather valve, the following procedure was employed:

a. A sensitive pressure recording instrument was installed to record the pressure inside the vapor tight compartment to determine the times that the internal pressure exceeded the breather valve setting.

b. The hydrocarbon content of the vapor space was determined both by mass spectrometer and J. W. Tester readings. It was found that the hydrocarbon content values by the two methods were in good agreement.

c. The flow rate (if any) was determined from the times the internal pressure exceeded the relief valve setting and the hydrocarbon emission was calculated using the analytical values.

To determine the vapor loss from the balance of the non-vapor tight compartments,the following procedure was used:

a. The volume per cent of hydrocarbons in the vapor space was determined by J. W. Tester readings.

b. The hydrocarbon loss was calculated from the hydrocarbon content and the vapor displaced per hour. It was assumed that the vapor in each compartment was totally displaced by air each hour. It was suggested that the rate of displacement could be established by injecting an inert gas into the vapor space of each compartment and determining its rate of disappearance.

TABLE XIV

METHOD 14C

Hydrocarbons and Hydrogen Sulfide Steam Ejector Vacuum Pumps

Sampling outlets were placed in the vertical standpipes on each tower, several feet above the point of discharge of the steam ejector into the line. The height of standpipe above the sampling outlet is approximately 50 feet in each case.

Sampling Procedure

Samples of each effluent stream were taken in three evacuated one-liter capacity glass bombs. The ejector discharge was allowed to blow through the short sampling tee tube until it was heated sufficiently to minimize steam condensation. Each bomb was then attached and filled for a 10-second period.

Analyses of Steam Ejector Effluents

Analyses and calculation of the weight compositions of the bomb contents were developed as follows:

1. The water content was determined by the difference in bomb weight when full and empty (at equal internal pressure) less the weight of condensed oil.

2. The weight of dry gas was determined by calculation from the absolute gas pressure in the bomb, the bomb volume, and the average molecular weight of the gas after correcting for temperature and the presence of water vapor.

3. The composition of the gas was determined by Podbielniak distillation (two bombs required) followed by a mass spectrograph of the C_5 and lighter fraction.

4. The oil content (condensed oil) of the bomb was obtained by washing out the bombs, after removing gas samples for Podbielniak distillation, with successive portions of saturated sodium sulfate solution. The oil content is determined by centrifuging the washings in a micro-Babcock bottle.

5. To check the results of the mass spectrograph for hydrogen sulfide, the contents of the third bomb, in each sampling, were extracted with sodium hydroxide solution and titrated potentiometrically with silver nitrate solution. In each case the mass spectrograph data agreed closely with the hydrogen sulfide content as determined potentiometrically. Hence, in future measurements the use of a third sample bomb would be unnecessary.

Determination of Steam Rates

The steam rates to the ejectors were calculated from design rates by correcting for changes in nozzle diameter, steam pressure, and degree of superheat. Calculations were also made to determine to what extent the steam would condense in the vertical standpipes in each case. These calculations indicated that due to the degree of superheat, after throttling through the ejector, little or no condensation of steam can occur.

Sample Calculation of Volume of Effluents

Calculation of the rate of discharge of effluents was made by multiplying the corrected ejector steam rate by the ratio of the weights of each contaminant to the weight of the accompanying water.

Sample Calculation of Steam Rates

	D-3 Ejector	D-9 Ejectors (2 ejectors in parallel)
1. Design Conditions		
a. Nozzle diameter	0.4724"	1
b. Steam rate	1,330 lb./hr.	1,850 lb./hr.
c. Steam conditions	130 psig. saturated	125 psig. 500°F.
2. Actual Operating Conditions		
a. Nozzle diameter	0.5312"	No change
b. Steam conditions	145 psig. 470°F.	145 psig. 470°F.

3. Determination of Current Steam Rate:

D-3 Tower

$$1{,}330\text{ lb./hr.} \times \frac{(145 + 14.7)}{(130 + 14.7)} \times \frac{(.5312)^2}{(.4724)^2} \times \frac{2.75\text{ (sp. vol. sat. steam)}}{3.25\text{ (sp. vol. }110^{\circ}\text{F. superheat)}}$$

= 1,600 lb./hr. steam rate

D-9 Tower

$$1{,}850\text{ lb./hr.} \times \frac{(145 + 14.7)}{(135 + 14.7)} \times \frac{3.44\text{ (sp. vol. }140^{\circ}\text{F. superheat)}}{3.25\text{ (sp. vol. }110^{\circ}\text{F. superheat)}}$$

= 2,100 lb./hr. steam rate

METHOD 15 A

Hydrocarbons from Pumps

The basic steps used in measuring the hydrocarbon vapor leakage from pump shaft seals are:

1. Enclose the area directly around the seal as air tight as possible.
2. Draw the vapors and air from this area for a measured length of time and measure the total volume withdrawn in that period.
3. Measure the hydrocarbon percentage of the vapors withdrawn so that the amount of hydrocarbon lost during the test period can be determined. In the case of pumps in gasoline service, the hydrocarbon is collected as a liquid in a graduated cylinger and measured directly.

The enclosure around the seal area is made of vinyl plastic sheeting held in place with Glyptal varnish and masking tape. On some pumps, it is only necessary to seal openings in the housing around the seal; on others a wire frame must be used to hold the plastic sheeting away from the shaft. For pumps with reciprocating shafts, a plate made of cardboard with a neoprene gasket is used to achieve as good a seal as possible around the shaft. In all cases the enclosure is made as small as possible.

The vapors and air are drawn from the enclosure with a vacuum pump and the volume is measured with a wet test meter. The hydrocarbon percentage is measured with a J. W. Combustible Gas Indicator with Range Multiplier Cock, the reading of the meter being multiplied by a factor to determine the volume percentage of the particular hydrocarbon being pumped.

In each test the enclosure is first purged by the vacuum pump for a period of about ten minutes. The rate of flow is adjusted so that a partial vacuum is created in the enclosure. This insures that all of the hydrocarbon vapors will be removed from the area during the test. The hydrocarbon percentage is checked in a surge bottle attached to the outlet of the wet test meter.

It is important that the test time be measured accurately and that the flow rate remain constant during the test.

On pumps in gasoline service the area is enclosed to keep evaporation at a minimum and the liquid collected in a graduated cylinder either at the drain line or inserted directly under the seal.

METHOD 16D

Hydrocarbons and Sulfur Dioxide from Acid Sludge Storage and Handling

a. A side stream of the gas from the fume system was drawn through two condensers and graduated traps, in series; the first were cooled in an ice bath and the second were cooled by a dry ice petroleum thinner slurry. The hydrocarbon, water, and SO_2 were condensed and the remaining non-condensible gases were measured by a wet test meter.

b. Hydrogen peroxide solution was added to each trap to oxidize SO_2 to SO_3.

c. The traps were allowed to warm up to atmospheric temperature and the volume of the hydrocarbon layer was read.

d. The sulfate in the water phase was determined gravimetrically as barium sulfate.

e. The total amount of hydrocarbon and SO_2 in the fume system was calculated from the total gas volume assumed to be that of the design figure. In this case the fume line passed to a boiler firebox where the hydrocarbons were incinerated.

METHOD 17D

Mercaptans, Hydrogen Sulfide, and Ammonia from Sour Water Strippers

Refer to the attached sketch for a typical example of water stripping operation. The method of gas sampling, determining of flow rates, and types of analytical tests are described as follows:

I - Samples (Refer to the attached sketch for sample points and sampling equipment.)

1. Water samples are obtained of streams 1, 2, and 3.

2. Gas sample, stream 4, is obtained in the following manner: A 500 ml. graduated cylinder is filled to the 250 ml. mark with fresh 20% NaOH solution and fitted with a two-hole stopper as shown. The head space in the graduate is evacuated. After purging the sample line on stream 4, the acidic gases are absorbed in the NaOH solution, with the inert materials collecting in the head space. The increase in volume of the NaOH solution is a rough indication of the size of sample obtained. The NaOH absorber is then cooled and after thorough mixing the graduate is inverted and brought to atmospheric pressure with a water leveling bottle. The volume of non-absorbed gas is then measured. The caustic volume remaining is measured and specific analyses obtained. The sum of the absorbed materials and non-absorbed gas is the total sample obtained.

II - Calculation of Flow Rates

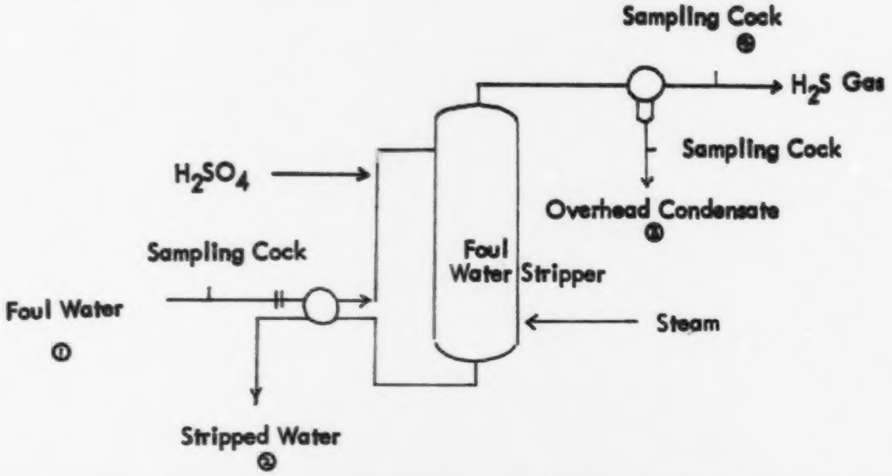
The foul water to the column is usually the only measured rate. However, the stripped water rate can be determined from the foul water rate by a heat balance, using temperature measurements on the bottoms to feed heat exchangers. The overhead condensate is estimated by checking the level increase per unit time in the overhead condenser water draw. The H_2S content of each of these three streams is determined. A material balance around the column yields the overhead H_2S rate, and with the analysis of the gas stream, the overhead gas rate.

III - Analytical Tests

The types of tests listed below are normally used; however, alternate methods are possible, particularly if interference from other materials is suspected.

SKETCHES FOR METHOD 17D TABLE XIV

SAMPLE STREAMS FOUL WATER STRIPPER


Sampling Cock
④
H_2S Gas
Sampling Cock
H_2SO_4
Overhead Condensate
③
Sampling Cock
Foul Water Stripper
Foul Water
①
Steam
Stripped Water
②

SAMPLING EQUIPMENT

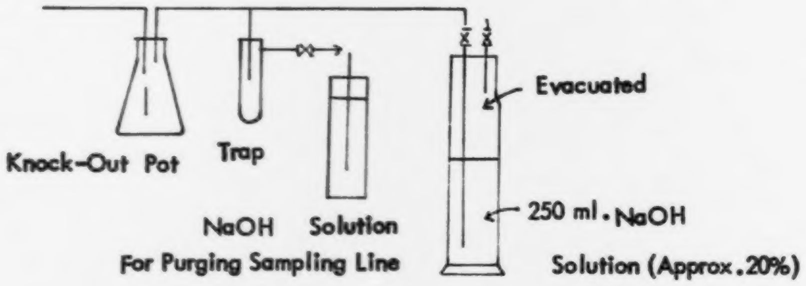

Evacuated
Knock-Out Pot
Trap
NaOH Solution
For Purging Sampling Line
250 ml. NaOH
Solution (Approx.20%)

Figure 13

1. H_2S - Iodometric titration.
2. NH_3 - Distillation from basic solution in Kjeldahl apparatus.
3. RSH - Electrometric titration or as an alternate the precipitation of H_2S and RSH with cadmium in neutral solution--filtered - titrated, followed by precipitation in slightly acid solution--filtered and titrated. The two step procedure separates the H_2S and RSH.

METHOD 20C

Hydrocarbons from Doctor Treating Units

General

The principal losses of hydrocarbon vapors at Doctor treating units occur in handling and regenerating spent Doctor Solution. The Doctor Solution (sodium plumbite), after contacting sour gasoline in the mixer, is separated from the entrained gasoline in one or more settlers. It is usually necessary to heat the Doctor Solution in order to break out the gasoline as a separate phase. Considerable evaporation of hydrocarbons occurs from the surface of the gasoline layer in the heated settler. After this separation of the entrained gasoline, the Doctor Solution is regenerated by blowing with compressed air in a regenerator vessel. The effluent air from this operation which contains traces of hydrocarbons is released to the atmosphere, usually after passing through a cyclone separator.

Evaporation Losses

Since there is no practical means of measuring evaporation losses directly, the loss of hydrocarbon vapors from the layer of gasoline in the settler is calculated from experimental data. The laboratory evaporation loss data were developed as follows:

> A known quantity of heavy cracked gasoline was placed in an open pan and heated on a hot water bath to a temperature of 150-160°F. A fan was placed so as to blow air across the face of the pan at a rate of 8 miles per hour, which corresponds to the average wind velocity in the refinery area. The loss in volume of gasoline was observed after a several hour period and expressed in gallons evaporated per hour per square foot of exposed surface.

Losses from Effluent Gases from the Doctor Solution Regenerator

A sample of the gases from the regenerator vessel discharge is obtained and analyzed in a mass spectrometer. From the analysis, the time of regeneration, and the average air rate to the regenerator, the loss from this source can be calculated.

PROCEDURES USED BY THE JOINT PROJECT FOR CHEMICAL ANALYSES OF SAMPLES COLLECTED

Analytical Procedure for Aldehydes[8]

Reaction

Sodium bisulfite adds to an aldehyde according to the following equation:

$$RCHO \; - \; NaHSO_3 \pm RCH\,(OH)SO_3Na$$

Reagents

1 per cent fresh sodium bisulfite solution, pH = 2.7
Starch indicator solution for iodimetric titration
Standard 0.1N iodine solution
Standard 0.05N sodium thiosulfate solution
Buffer solution: 80 g. of Na_2CO_3, 20 ml. of glacial acetic acid made up to a liter with distilled water, pH = 9.6

Procedure

After the sample is taken in the 2 liter evacuated flask containing 10 ml. of the sodium bisulfite solution, it should be allowed to stand for at least 20 minutes. The contents are then transferred to a 250 ml. conical flask. Add 2 ml. of starch indicator solution and 0.1N iodine solution, just sufficient to obtain a dark blue color. Carefully wash down the sides of flask. Decolorize by adding a drop or two of 0.05N sodium thiosulfate solution and bring to faint blue color with 0.005 N iodine.

Chill thoroughly in an ice bath, add 50 ml. of thoroughly chilled buffer solution and allow to stand in ice bath for 10 minutes. Titrate the liberated bisulfite with 0.005N iodine to the same faint blue color end point.

Run a blank using the same quantities of reagents and subtract it from the titration.

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[8] Goldman, F. H., and Yagoda, H., "Collection and Estimation of Formaldehyde in the Air," Ind. and Eng. Chem. (Anal. Ed.) 15, 377 (1943)

Calculations

The sample volume is corrected to 60°F. and 760 mm. Hg.

$$\text{PPM as HCHO} = \frac{(\text{ml. of } I_2)(N) \times 11.8 \times 10^3}{\text{corr. vol. in liters}}$$

N = normality of iodine solution

Note

The efficiency of this method for the determination of aldehydes decreases as higher homologs are encountered.

Analytical Procedure for Ammonia[9]

Reagents

30 per cent sodium hydroxide solution
Standard 0.1N sulfuric acid solution
Standard 0.02N sodium hydroxide solution
Methyl red indicator solution

Procedure

Combine the filtered solutions from the three impingers from the water-HCl sampling train and note the volume. Dilute a 50 ml. aliquot of the sample to approximately 250 ml. in a 500 ml. Kjeldahl flask. Add a few glass beads or boiling pellets to prevent bumping of the solution. Add 25 ml. of 30 per cent sodium hydroxide solution and quickly connect the flask to a Kjeldahl distilling rack.

Distil 100-150 ml. of the solution into a 500 ml. Erlenmeyer flask containing an excess (100 ml.) of 0.1N sulfuric acid. The delivery tube must dip below the surface of the 0.1N sulfuric acid, and care must be taken to present the solution from "sucking back" into the Kjeldahl flask.

Titrate the excess sulfuric acid with 0.02N sodium hydroxide, using four drops of methyl red as an indicator.

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[9] Scott, "Standard Methods of Chemical Analysis," 5th Edition, Vol. I, pp. 637-63

Note

The amount of solution taken as an aliquot may be varied, depending upon the concentration of ammonia present.

Calculations

$$\text{grams of ammonia collected in train} = \frac{(100\,N_1 - V_3N_2)\,(V_1)\,(0.017)}{(V_2)}$$

where N_1 = normality of sulfuric acid solution

N_2 = normality of sodium hydroxide solution

V_1 = total volume of solution in impingers

V_2 = volume of aliquot taken for analysis

V_3 = volume of sodium hydroxide solution used in titration of excess sulfuric acid

Analytical Procedure for Cyanides[10]

Reagents

15 per cent hydrochloric acid solution
Saturated lead acetate solution
1 per cent picric acid solution. Commercial picric acid usually contains about 10 per cent water, and this must be considered when the solution is made up.
Potassium dichromate crystals
Standard potassium cyanide solution equivalent to 100 mg. HCN per liter (standardized)
Sodium carbonate solutions; 0.25 molar, 0.5 molar, and 1 molar
10 per cent sodium hydroxide solution
15 per cent sulfuric acid solution
Methyl red indicator

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[10] "Determination of Hydrogen Cyanide and Cyanogen," Colorimetric Method, API Method 765-54, Manual on Disposal of Refinery Wastes, Vol. V., "Sampling and Analysis of Waste Gases and Particulate Matter," first edition.

Procedure

A 100 ml. aliquot of the stack sample (absorbed in 5 per cent sodium hydroxide solution) is transferred to the flask of a distillation apparatus as illustrated in Fig. 765.1, API Manual on Disposal of Refinery Wastes, Volume V. Volatile reducing agents, notably sulfites and sulfides, interfere with the determination, so the absorber solution must be tested for the presence of these substances as follows:

> Sulfides: Test the absorber solution by placing a drop on lead acetate test paper or by some equivalent method. If sulfides are present, proceed as described under "Sulfides Present."
>
> Sulfites: Mix a drop of 10 per cent potassium ferrocyanide solution, a drop of cold saturated zinc sulfate or zinc nitrate solution, and a drop of 1 per cent sodium nitroprusside solution. A white precipitate of zinc ferrocyanide is formed. To this add a drop of neutralized absorber solution. If the precipitate develops a red color, sulfites are present. In this case proceed as described under "Sulfites Present."

A. No sulfides or sulfites present: Add a few drops of methyl red indicator solution to the solution in the distillation flask and connect the flask to the distillation apparatus.

Immerse the sintered glass tip of the condenser tube in 25 ml. of 1 molar sodium carbonate solution contained in a 100 ml. graduated cylinder. Apply a low flame to the distillation flask, and when boiling has started, pass steam through the system. When reflux has started and back pressure has caused the carbonate solution to rise slightly in the condenser tube, add slowly from the dropping funnel 15 per cent by weight sulfuric acid until the sample is distinctly acidic to methyl red indicator. If the sample contains a large amount of carbonate or bicarbonate, large quantities of carbon dioxide will escape from the system. This evolution will cease when the methyl red indicator changes color, and again the rapid condensation of the steam will cause a back pressure in the system and the carbonate solution in the cylinder to rise slightly. This back pressure should be maintained throughout the remainder of the distillation. Negligible hydrogen cyanide is lost during the evolution of carbon dioxide.

When the distillate volume has reached about 75 ml., lower the receiving graduate and increase the heat to the distillation flask to provide for a thorough washdown of the inside of the condenser by the condensate. Wash down the outside ot the condenser and stop the distillation.

Transfer this distillate to a 100 ml. volumetric flask and dilute to volume with distilled water. Pipette a 10 ml. ± 0.01 ml. aliquot into a dry

100 ml. volumetric flask and add 5 ml. ± 0.01 ml. of the picric acid solution. Develop the color by immersing the flask in boiling water for 5 min. ± 0.5 min. Remove and fill almost to the mark, cool to room temperature, and fill exactly to the mark.

Observe the absorption at 520 mμ against distilled water in a photometer. If the absorption is greater than that covered by the standard curve, transfer a smaller aliquot portion of the distillate to a 100 ml. volumetric flask and add sufficient 0.25 molar sodium carbonate solution to bring the volume to 10 ml. ± 0.01 ml. Then add 5 ml. ± 0.01 ml. of picric acid and repeat as outlined hereinbefore.

B. Sulfites Present: Add a large excess of potassium dichromate crystals (1 g. is usually sufficient) to the absorber solution in the distillation flask to oxidize all sulfites present. Add a few drops of methyl red indicator solution, attach the flask to the distillation apparatus, and proceed as described in A, "No sulfides or sulfites present."

C. Sulfides Present: After placing the absorber solution in the distillation flask, cool the flask in an ice bath and titrate the solution to a pH of 2 with a glass electrode or indicating paper using 15 per cent hydrochloric acid solution. Add enough saturated lead acetate solution to precipitate all of the sulfide present, and then add 2 ml. in excess. Stopper the flask and shake vogorously to insure the removal of any hydrogen sulfide above the solution.

Attach the flask to the distillation assembly and pass steam into the cold solution. Continue the distillation as described in A, "No sulfides or sulfites present," omitting the addition of sulfuric acid. Test a portion of the distillate for sulfide with a drop of lead acetate solution. If the test is positive, either the sulfide precipitation is incomplete or the pH of the solution is too low. Repeat the procedure on a fresh portion of the sample, making appropriate corrections.

Preparation of Standard Curve

Pipette 10, 20, 50, and 100 ml. portions of the standard potassium cyanide solution into separate 100 ml. volumetric flasks and dilute each to volume with distilled water.

Pipette a 5 ml. ± 0.01 ml. aliquot from each flask into dry 100 ml. volumetric flasks. Prepare a blank by pipetting 5 ml. ± 0.01 ml. of distilled water into a fifth volumetric flask. To each of the 5 flasks add 5 ml. ± 0.01 ml. of the 0.5 molar sodium carbonate solution and 5 ml. ± 0.01 ml. of the 1 per cent picric acid solution, in that order.

Immediately immerse the flasks in boiling water for 5 min. ± 30 sec. Remove the flasks from the boiling water and, to stop any further reaction, dilute with distilled water at once to slightly less than final volume. Cool the flasks to room temperature in tap water and make a final adjustment to volume. The developed color is stable for several hours.

Determine the absorption at 520 mμ of the developed color of each solution against distilled water in a photometer. The absorption of the picrate blank should be very low and constant (< 0.004) at this wave length.

Prepare a graph of absorption against equivalent hydrogen cyanide concentrations of 0.05, 0.10, 0.25, and 0.50 mg. per 100 ml. The absorption of the picrate blank need not be used in the graph. If the procedure has been followed carefully, the standard curve will be a straight line.

Calculation

Read the hydrogen cyanide concentration from the standard curve for the absorption obtained in the analysis. Calculate the amount of cyanides in the sample gas by the following equation:

$$\text{Cyanides, per cent by volume as HCN} = \frac{77.2E}{BDV}$$

where E = hydrogen cyanide, milligrams, read from standard curve

B = volume of aliquot taken from distillate, milliliters

D = volume of aliquot taken for distillation, milliliters

V = volume of gas sample, cubic feet, at 60°F. and 1 atm.

$$77.2 = \left[\frac{100\ \text{(ml. of dist.)}}{1000\ \text{(mg. per g.)}}\right]\left[\frac{23.6\ \text{(liters per mol)}}{27\ \text{(M.W. of HCN)}}\right]\left[\frac{250\ \text{(ml. of absorber solution)}}{28.3\ \text{(liters /cu. ft.)}}\right]\quad 100(\%)$$

Analytical Procedure for Organic Acids[11]

Reagents

Distilled ether
Conc. fulfuric acid
Standard 0.1N sodium hydroxide solution

Procedure

A 100 ml. aliquot of the combined impinger solutions from the sodium hydroxide train is placed in a 500 ml. stoppered Erlenmeyer flask. The sample is then chilled in an ice bath. A 100 ml. volume of distilled ether is added to the sample. Concentrated sulfuric acid is added slowly with constant stirring to a pH of 2 to 3 (test with pH paper). The addition of sulfuric acid releases the free organic acid which is miscible with ether.

The mixture is removed to a separatory funnel and thoroughly shaken to remove the organic acid from the water layer. The ether layer is preserved, while the water layer is shaken four more times with ether, each time with a 100 ml. portion of ether. The ether extracts are combined and the original water layer is discarded.

The organic acids in the ether extract are now titrated with 0.1N sodium hydroxide solution using phenol red indicator. Prior to titration, 75 ml. of distilled water are added to the ether extract. The addition of water is necessary in order to dissolve the sodium salts of the organic acids.

The water layer is separated and the ether is washed three times with 35 ml. portions of distilled water. The water washings are combined and titrated with 0.1N sodium hydroxide solution.

Calculations

$$\text{mg. of organic acids, as acetic acid} = \text{ml. of sodium hydroxide solution} \times N \times 60 \times \frac{V}{V_a}$$

N = normality of sodium hydroxide solution used in titration
60 = molecular weight of acetic acid
V = volume of sample submitted
V_a = volume of aliquot

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[11] "Analytical Procedure for Organic Acids," APCD Research Division Methods

Analytical Procedure for Oxides of Nitrogen[12]
(Phenoldisulfonic Acid Method)

Reagents

Absorbing Solution:

An absorbing solution is prepared by adding 1 ml. of 3 per cent hydrogen peroxide to 100 ml. of 0.1N sulfuric acid. Twenty-five ml. of this freshly prepared absorbing solution is added to each of the 2 liter sample flasks and about 50 ml. is retained for running solution blanks.

Phenoldisulfonic Acid Solution:

Dissolve 25 g. of pure phenol in 150 ml. of concentrated sulfuric acid on a steam bath, cool and add 75 ml. of fuming sulfuric acid (15 per cent SO_3). Heat to 100°C. for 2 hours. Store in a brown, glass-stoppered bottle. This solution will be colorless if prepared from pure reagents. It will deteriorate on long standing and yield a high blank.

1 N (approx.) sodium hydroxide solution
conc. sulfuric acid
conc. ammonium hydroxide

Procedure

The sample is allowed to stand at least 6 hours to insure complete absorption of the oxides of nitrogen. When absorption is complete, all oxides of nitrogen with the possible exception of nitrous oxide (N_2O) will be converted to nitric acid. The following reactions are involved in the case of nitrogen dioxide:

$$2NO_2 + H_2O = HNO_2 + HNO_3$$

$$HNO_2 + H_2O_2 = HNO_3 + H_2O$$

The contents of the 2 liter sampling flask are transferred quantitatively to a 200 ml. casserole, and the solution is made alkaline to litmus with 1N sodium hydroxide solution.

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[12] "Analytical Procedure for Oxides of Nitrogen, Phenoldisulfonic Acid Method," APCD Research Division Methods

A blank is prepared by placing 25 ml. of original absorbing solution in a casserole, diluting to approximately the same volume as the sample and making alkaline to litmus. The solutions are then evaporated to dryness on a steam bath.

Cautiously add 1 ml. of phenoldisulfonic acid solution to the dry residue and triturate thoroughly with a glass rod. Add 1 ml. of water and 4 drops of concentrated sulfuric acid. Heat on a steam bath for 3 minutes with occasional mixing. Cautiously add 25 ml. of water followed by 5 ml. of concentrated ammonium hydroxide. A yellow color is developed. Filter the solution and dilute to 50 ml.

The light absorption of the solution is measured in a Klett-Summerson photoelectric colorimeter using a blue (No. 42) light filter and a 20 mm. light path. The colorimeter may be zeroed against the blank, or against distilled water and the blank value subtracted from each reading. The weight of nitrogen dioxide in the sample is obtained from a standard colorimeter chart prepared from known standard potassium nitrate solutions.

Calculations

mg. of nitrogen dioxide per standard cubic foot $= \frac{W \times 28.3}{V_c \times 10^3}$

p.p.m. (vol.) of nitrogen dioxide $= \frac{W \times 23.7 \times 10^6}{46\, V_c}$

where W = weight of nitrogen dioxide in grams
V_c = volume of sample in liters corrected to 760 mm. Hg and 60°F.

Notes and Comments

1. The more concentrated samples may yield colors too dark to be read on the colorimeter. The sample may be diluted with water. An alternative is to use a smaller sampling flask.

2. Since hydrogen peroxide is necessary to change the nitrite to nitrate, an adequate amount is necessary. The amount should always be in excess over oxidizable material in the sample.

3. Inorganic nitrates or organic nitrogen compounds which are easily oxidized will interfere with this method, giving high results.

4. The sensitivity of this method has been found to be about 5 p.p.m.

Analytical Procedure for Oxides of Sulfur

Reagents

Bromine water
Concentrated hydrochloric acid
10 per cent barium chloride solution
Silver nitrate solution

Procedure

Dilute a 100 ml. aliquot of the combined impinger solutions from the 5 per cent sodium hydroxide sampling train to 200 ml. Add bromine water in slight excess (until color remains) to oxidize the SO_2. Boil until the solution is colorless and continue boiling for another five minutes to expel the excess bromine. Neutralize with concentrated hydrochloric acid and add 2 ml. excess. Filter if necessary to remove any silica or other solid material. Heat to boiling and add 10 per cent barium chloride solution with constant stirring until an excess is present. Digest the solution for one hour over a steam bath. Filter through a fine ashless filter paper, checking the filtrate for complete precipitation. Wash with hot water until the filtrate is free of chloride as determined by adding silver nitrate solution to the washings. Transfer the paper and precipitate to a tared crucible, dry and ignite to constant weight at 750°C.

The water extract of sulfur trioxide from the Whatman thimbles is determined as above with the exception that bromine oxidation is omitted.

A

PETROLEUM STORAGE TANK

COMPANY:

LOCATION OF TANKS:

TANK NUMBER OR DESIGNATION	TANK TYPE	TANK SHAPE	TANK CAPACITY (BBLS.)	DIMENSIONS		TYPE SHELL CONST.	COLOR PAINT OR INSULA. ON SHELL	TANK MECH. CONDIT.	TYPE OF ROOF	TYPE OF F.R. SEAL	PRESS. VENT SET-TINGS	WHERE VENTED	REMARKS.		PRODUC
				DIAM. OR LENGTH (FT.)	HT. OR WIDTH (FT.)										

TANK TYPE

C = Fixed or Coneroof
F = Floating Roof
P = Pressure

TANK SHAPE

VC = Vertical Cylind.
HC = Horiz. Cylind.
S = Spherical or Spheroidal
R = Rectangular

TYPE SHELL CONSTR.

W - Welded
R = Riveted
RG = Riveted and Gunited
B = Bolted

TYPE OF ROOF

C = Fixed or Cone
W = Water Seal
O = No Roof, Open Top
D = Doubledeck F.R.
P = Pontoon F.R.
PM. = Pan F.R. Modified
Pan = Pan F.R.

TYPE F.R. SEAL

O = No Seal
S = Single Seal
D = Double Seal
T = Tube Seal

COLOR PA

CW = Ch
AL = Al
LG = Lig
B = Dar
or
IN = In

AGE TANK and PRODUCT DATA

D

TANKS: DATE:

MARKS.	PRODUCT	VAPOR PRESSURE EITHER RVP (LBS)	OR ABS. (PSIA)	AT TEMP (°F)	FILL IN ONLY IF VAPOR PRESSURE DATA NOT AVAILABLE FOR PRODUCT FLASH PT (°F)	IBP (°F)	FBP (°F)	API GRAVITY	AVG. LIQUID BODY STORAGE TEMP (°F)	MAX. LIQUID BODY STORAGE TEMP (°F)	MAX. FILLING RATE (BBL/HR)	AVG THROUGHPUT (BBL/DAY)	TURN-OVER PER YEAR	AVG OUTAGE	

E F.R. SEAL

No Seal
Single Seal
Double Seal
Tube Seal

COLOR PAINT OR INSULATION

CW = Chalking White
AL = Aluminum
LG = Light Gray
B = Dark Color, No Paint or Needs Repainting
IN = Insulated

TANK MECH. CONDITION

G = Good (vapor tight)
F = Fair (minor leakage)
P = Poor (needs repair)

WHERE VENTED

A = Atm.
V.R. = Vapor Recovery
V.D. = Vapor Disposal
V.B. = Vapor Balancing

REMARKS (EXAMPLES)

Micro Balloon
Equipped for Air Blowing
Equipped with Heating Coils
Open Vents
Etc.

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